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The Chemical Contouring of 3% Chromium-Molybdenum-Vanadium and 5% Chromium-Molybdenum-Vanadium High Strength Steel Sheet

By

Bristol Aerojet Ltd.

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The Chemical Contouring of 3% Chromium-Molybdenum-Vanadium and 5% Chromium-Molybdenum-Vanadium High Strength Steel Sheet

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1. Summary

Earlier development of a chemical contouring process for 1% chromium-molybdenum steel sheet was described in S. & T. Memos 20/60 and 23/60. The present report describes the development of processes for 3% chromium-molybdenum-vanadium steel sheet and 5% chromium-molybdenum-vanadium steel sheet both heat treated to tensile strengths of not less than 105 tons/sq.in. A common etchant solution for all three steels was also developed.

Preferred etchant compositions and proprietary masking materials are given. Etching must be carried out within fairly closely defined limits of temperature.

Specimen panels of both steels have been prepared. The surface finish was good and edge definition acceptable. All panels, however, showed considerable variation in etch depth, the depth being greater at the centres of etched pockets than near the edges.

2. Introduction

Chemical contouring can be described as the removal of metal by controlled chemical action. The areas of metal not to be removed are protected by a surface coating of an inert substance, usually a lacquer, termed a maskant. The process is particularly useful for removing metal for weight saving from sheet components and can be applied after the components have been formed and heat-treated. Mechanical milling of such components would be difficult, especially if the metal is strong and hard.

The maskant can be applied to the metal surface be either dipping, spraying or brushing. It is normal to apply more than one coat of lacquer and each coat is allowed to dry before the application of the succeeding coats. When a predetermined thickness of coating has been obtained the maskant is heated or stoved at a recommended temperature for a given period of time. Stoving conditions are specific for each lacquer. The pattern required is then scribed on the surface of the maskant and maskant removed to expose the areas of the material which are to be chemically contoured.

For low alloy steels the etchant is an acid or a mixture of acids in which the specimen is immersed. As expected, a rise in temperature will accelerate the rate of chemical attack on the metals; for low alloy steels a suitable etching rate is achieved by etchants at about 60 - 70°C.

When developing an etchant and a maskant the desirable characteristics of the etched specimen are: uniform attack, smooth surface finish, and good edge definition. The undesirable side effects of channelling, striations, scalloping, valleying, double edge and excessive undercutting should be avoided. These and other defects are illustrated in Figs. 1 to 9.

The etchant must show a reasonably high etch rate, (ca 0·060 in/hr.) long etchant life, stability at operating temperatures and passivation of the specimen must not occur. The life of an etchant can be defined as the maximum amount of iron that can be dissolved in the etchant without causing unacceptable deterioration in etch rate and in the surface finish and edge definition of the

etched/

etched area. As the etchant ages the etch rate decreases. The rate can be increased again by the addition of predetermined volumes of fresh acid (or acids) but the regenerated etchant does not necessarily give the same surface finish and line definition, both of which usually deteriorate towards the end of the etchant life.

The maskant must be completely inert to the etchant and must strip easily from the specimen after contouring. During the contouring investigation the maskant was generally reinforced at the specimen edges by the use of polyester, silicone adhesive tape.

Techniques developed by Bristol Aerojet Limited for the chemical contouring of 1% chromium-molybdenum steel have been described in Ministry of Aviation S. & T. Memos 20/60 and 23/60.

This report covers the development of etchants and maskants for 3% chromium-molybdenum-vanadium and 5% chromium-molybdenum-vanadium steels in sheet form. The report is divided into sections (see list of contents) each section being self contained. The results are discussed at the end of each section and conclusions are stated. A general discussion is included towards the end of the report with conclusions appertaining to the whole investigation. Dimensions of specimen contoured test panels are detailed and brief mention is made concerning the costs of chemical contouring. Sustained load and fatigue tests on contoured specimens are being made and will be reported separately. Definitions of contouring terms used in the report are given in the Appendix.

3. Etchants and Maskants for 3% Cr-Mo-V Steel Sheet (RS.140)

3.1 Material

Steel sheet to Bristol Aerojet Specification RS.140 was used. This internal specification has requirements lying within, and closer than, those of En.40C; in particular, the sulphur and phosphorus are low.

Sheets were obtained from Burys and Co. (batches BU 334, 514, 515 and 677), J. J. Habershon and Sons Ltd. (batches HAB 35 and 36), Jessop-Saville Ltd. (J.14) and Marathon Fine Steels Ltd. (DEW.3). The chemical compositions of these materials, and the limits of RS.140 are given in Table I.

Table I/

Table I

Chemical composition of RS.140 3% Cr-Mo-V steel sheet materials

Batch mark	Gauge	Composition, % by weight								
		C	Mn	Si	S	P	Ni	Cr	Mo	V
BU 334	10	0.40	0.68	0.26	0.006	0.005	0.10	3.27	0.91	0.24
BU 514	8	0.41	0.66	0.27	0.005	0.009	0.10	3.25	0.95	0.26
BU 515	12	0.42	0.68	0.26	0.003	0.009	0.10	3.27	0.91	0.24
BU 677	10	0.43	0.64	0.26	0.008	0.011	0.09	3.02	0.94	0.21
HAB 35	15	0.42	0.70	0.26	0.010	0.010	0.20	3.19	1.00	0.21
HAB 36	15	0.37	0.68	0.27	0.010	0.010	0.16	3.17	1.00	0.19
J 14	13	0.42	0.57	0.38	0.006	0.012	0.37	3.17	0.80	0.22
DEW.3	15	0.40	0.67	0.28	0.010	0.012	-	3.00	0.98	0.20
RS 140 limits		0.38-0.43	0.50-0.70	0.10-0.35	0.010 max	0.015 max	0.30 max	2.9-3.3	0.90-1.10	0.15-0.25

Initial specimens from Batches HAB.35, HAB.36 and J.14 were oil quenched from 920°C and tempered at 540°C., but it was found that the microstructure produced by this treatment was not always homogeneous, resulting in a wide range of hardness on individual specimens. This variable was established as a major factor influencing etch rate variation and surface texture. Consequently, the austenitizing temperature was increased to 940°C. for all later work. This heat treatment produced improved texture of the etched surfaces and less variation in etch rate. The hardness scatter range was reduced from 440-520 HV. to 490-510 HV. indicating a more uniform structure. The equivalent tensile strength range is 105-109 tons/sq.in.

Some specimens from Batches BU.514 and BU.677 were martempered by austenitizing at 940°C. for 40 min., cooling to 510°C. and holding for 60 min., quenching in Edgar Vaughan's 2E oil, and tempering at 300°C. for 60 min. This treatment produced hardnesses equivalent to a tensile strength range of 116-122 tons/sq. in.

3.2 Development of etchant for 3% Cr-Mo-V steel sheet

3.2.1 Materials used

Most of the work and all the final evaluation, was done on batches BU.514 and BU.677 martempered.

3.2.2/

3.2.2 Surface preparation

Rough surface specimens used during initial work were pre-etched in a 12% nitric-5% sulphuric acid solution at 60°C., followed by linishing. This treatment was necessary to avoid uneven surface finishes after contouring. With the etchant finally chosen, pre-etching and linishing were unnecessary.

The surfaces of all the specimens were degreased in trichlorethylene vapour and the heat treatment scale was removed by pickling in Ferroclene 100, an inhibited phosphoric acid/hydrochloric acid pickle. The samples were finally washed in water and swabbed prior to masking.

3.2.3 Initial etchant development

Initial etchant trials were carried out with the 18% sulphuric acid - 5% nitric acid - water etchant at 70°C developed for the 1% chromium-molybdenum steel. This acid mixture was found unsuitable for RS.140 steel due to a slow etch rate and unacceptable surface finish. Trials with other high sulphuric acid content etchants showed these to be unusable because of channelling and passivation of the steel surface, and 5% sulphuric acid etchants with varying additions of nitric acid gave unreproducible results.

Tests with hydrochloric acid - sulphuric acid and hydrochloric acid - nitric acid mixtures gave unsatisfactory results due to poor surface finish. The use of hydrochloric acid has the added disadvantage of the greater likelihood of plant corrosion.

The most encouraging results were obtained using a nitric - sulphuric - phosphoric acid etchant, the best composition of which was found to consist of 12% nitric acid, 5% sulphuric acid, 5% phosphoric acid in water. Phosphoric acid is considered to exert a surface smoothing effect and a reaction rate control on chemical and electrolytic etching and polishing processes.

Specimens from HAB.36 were masked with either Docker's 3:2 Stop-off: Strip-off mixture or Cellon's P2/50 lacquer and, after cutting a rectangular area from the maskant, etched in the 12-5-5 acid mixture at 70°C. Results showed that the surface finish was acceptable but the line definition poor. Channelling and under-cutting were not evident but the fact that the etch rate was initially 0·048 in/hr. at an iron concentration of 8·5 gm/litre, falling rapidly to 0·020 in/hr. at 20·0 gm/litre, led to the abandoning of this etchant.

It had been found from previous work on contouring that nitric acid plays a major part in the dissolution of low alloy steels. On this count it was decided to pursue the possibility of nitric acid alone being a suitable etchant for RS.140 steel. In order to evaluate a 20% nitric acid in water etchant, specimens were obtained from Batch HAB.35 in the form of panels 3 in. × $1\frac{1}{2}$ in. × 0·120-0·135 in. By weighing and measuring before and after

contouring/

contouring the specimens for a predetermined period of time, the etch rate in relation to iron concentrations were determined. The first experiments were made at 77°C.

An overall etch depth of about 0•100 in. was stipulated and the etchant was regenerated when the etch rate dropped below 0•040 in./hr. The regeneration was carried out four times to give a useful etchant life of 50 gm./litre dissolved iron. Generally the surface finish was good but channelling was invariably present. The etch rate was erratic, varying from 0•100 in./hr. with the new etchant and, after regenerating, to a maximum of 0•135 in.hr., then to a minimum of 0•040 in./hr. prior to regeneration.

As the etchant gave such a diversified etch rate at 77°C., contouring was carried out with the acid temperature maintained at 70°C. At this temperature the etch rate varied between 0•045in./hr. and 0•080 in./hr. but channelling was very pronounced. Furthermore many specimens were eaten through around the edges whilst still showing a considerable thickness at the centre. The surfaces, although smooth, tended to undulate and passivation of the metal was often evident.

In spite of satisfactory surface finish and etchant life characteristics, the 20% nitric acid etchant was considered to be unsuitable for the contouring of RS.140 steel on account of excessive channelling and the passivation phenomenon.

3.2.4 Development of 20% nitric acid - 5% phosphoric acid as an etchant

As the 20% nitric acid etchant had proved to be satisfactory in some respects it was decided to evaluate 20% nitric acid with a 5% phosphoric acid addition.

The etching was carried out at 70°C. and exceptionally good surface finishes were obtained. (Figure 12). After regeneration with a mixture of phosphoric acid and nitric acid the surface tended to become striated and undulating, but if nitric acid alone was added these adverse effects were minimized. (Figure 13). Channelling was not experienced except with certain types of maskant. The initial etch life was found to be 30 gm/litre and a regenerated acid gave good results at concentrations of 50 gm/litre dissolved iron. Regeneration was effected by the addition of nitric acid.

As with the results with the 20% nitric acid etchant, the etch rate was found to vary between 0•160 in./hr. and 0•045 in./hr. This range was considered to be excessive and further work was carried out on the etchant maintained at 60°C. and 65°C. with encouraging results.

It was finally concluded that this etchant can be used for contouring RS.140 steel provided that the temperature of the etchant is held at 60°C until the iron concentration is 12 gm/litre. The

temperature/

temperature is then raised to 65°C until 20 gm/litre is reached and thence to 70°C. until the etchant reaches the end of its natural life at 30 gm/litre. Using this procedure the etch rate is maintained between 0·050 and 0·070 in./hr. (Figure 14).

3.2.5 Discussion

The etchant initially developed for RS.140 steel (i.e. 12% nitric - 5% phosphoric - 5% sulphuric acids) gave fairly satisfactory specimen results but its short life and the difficulty of regeneration of a ternary mixture make it an uneconomical proposition. Another limitation is that varying surface finishes affect the results, and time-wasting pre-etching and, sometimes, additional linishing treatments are necessary.

The 20% nitric acid etchant gave reasonable results but the unpredictable occurrence of passivation and the occasional rather severe channelling make the etchant unreliable and, in consequence, unsatisfactory for general use.

The 20% nitric acid - 5% phosphoric acid - water etchant gave good results. The quality of the surfaces of the specimen was high, and grooving, although becoming evident as the etchant aged, initially was completely absent. Line definition was good and under-cutting negligible. It is also important to note that as compared with the 12:5:5 etchant, both the 20 and the 20:5 etchants required considerably less surface preparation of the steel samples.

The effect of the varying composition from one batch of steel to another on the etch rate has not been studied in detail. The results in Figure 12, obtained at 70°C, suggest that with fresh etchant there may be a considerable variation in etch rate from one batch of steel to another. As the iron concentration in the etchant increases the etch rate variation tends to decrease. When contouring a component it is necessary to measure the etch depth after a known time and then calculate the further time required to etch to a specified depth.

3.2.6 Conclusions

An etchant composed of 20% nitric acid + 5% phosphoric acid is suitable for the chemical contouring of RS.140 steel provided that the specified etchant temperature is adhered to as the iron concentration of the etchant increases (i.e. 60°C. to an iron concentration of 12 gm/litre, 65°C. to a concentration of 20 gm/litre and 70°C. to regeneration at 30 gm./litre.).

Good surface finish and line definition were achieved and under-cutting was negligible. The etch rate lay between 0·050 and 0·070 in./hr. The etch rate tends to vary from batch to batch of the steel.

3.3 Evaluation of maskants for 3% Cr-Mo-V steel sheets

3.3.1 Materials used

Materials from batches BU.334, 514, 515, HAF.35 and DEW.3 were used. Most were quenched and tempered as described in Section 3·1,

but/

but some were martempered and are specifically mentioned as such below.

3.3.2 Surface pretreatment

The heat treatment scale was removed by acid pickling in Ferroclene 100 followed by immersion in 'Collex 304' alkali cleaner. In special cases linishing or pre-etching was adopted as an alternative method and where these methods were used special mention will be made.

3.3.3 Preliminary maskant evaluation

Maskants were evaluated for three of the etchant compositions described in Sections 3.2.3 and 3.2.4. These were:-

(a) 12% nitric acid, 5% phosphoric acid, 5% sulphuric acid.

(b) 20% nitric acid.

and (c) 20% nitric acid, 5% phosphoric acid (the etchant finally chosen.)

For brevity these etchants will be referred to as 12:5:5, 20 and 20:5, respectively.

3.3.4 Maskant application

The maskants were applied to the specimens by dip coating, allowing each coat to dry before applying the next. The thicknesses of coatings varied and the lacquers were finally stoved at a temperature recommended by the manufacturer, normally prior to the pattern being scribed.

3.3.5 Maskant evaluation in the 12:5:5 etchant

Table II shows the complete range of maskants used to protect RS.140 steel during contouring in the 12:5:5 etchant.

Table II/

Table II

Maskants Evaluated in the 12:5:5 Etchant

Neoprene Type	Vinyl Type	Miscellaneous
British Paints PR.785 PR.787 PR.790 Richardson's CER.4E Docker's Black Lacquer TGX.69	Dockers Strip Off Lacquer (Heavy Bodied) Cellon's Birlon SL6098 -do- No.2 Clear -do- No.3 Blue -do- H17 89L -do- Q7 14A -do- Q7 17A John Halls Transparent Amber 8/949/444. John Halls' Liq.Green Envelope -do- Liq.Blue Envelope 8/949/233 -do- Liq.Blue Envelope 8/949/252	Docker's Stop Off Lacquer Docker's 3:2 Stop Off Strip Off Lacquer Richardsons Adcora Hypalon H2. Croda Ltd. Rhinohide.

Overall observations indicated that for the majority of the lacquers, fresh solutions of etchant gave consistently good results. Exceptions were found in the Neoprene Group (Richardsons CER.4E), Vinyl Group (Cellon's Birlon SL.6098, No. 2 Clear and No. 3 Blue) and Miscellaneous (Docker's Stop Off Lacquer and Richardson's Adcora Hypalon H2).

However, it was found that using specimens of RS.140, Batch No. BU.515, although a fresh solution of the etchant initially gave good edge definition, satisfactory results were not maintained. It was noticed that as the etchant aged (or the dissolved iron concentration increased) the edge definition became poorer until at an iron concentration of only about 12 gm/litre of etchant, the results became unacceptable. The etch rate also deteriorated and regeneration of the etchant, although temporarily increasing the etch rate, did not bring about an improvement in the edge definition. The rapid deterioration of both etchant life and line definition led to the abandoning of this etchant.

3.3.6 Maskant evaluation in the 20 etchant

A number of maskants were evaluated, acceptable results being obtained using material from Batch HAB.35 masked with the Liquid Blue Envelope 8/949/252, Docker's 3:2 Stop Off Strip Off and John Hall's Transparent Amber lacquers. Further work on Batch BU.334, using the same three lacquers, revealed channelling, excessive eat-back under the maskant, and passivation. Both batches of steel

were oil quenched and tempered. On account of these results, further work involving this etchant and maskants for it was suspended.

The maskants evaluated are shown in Table III.

Table III

Maskants Evaluated in the 20 Etchant

Neoprene Type	Vinyl Type	Miscellaneous
Richardson's CER.4E Docker's Black Lacquer TGX.69	Docker's Strip Off Lacquer (Heavy Bodied) Cellon's Birlon SL 6098 -do- No. 3 Blue -do- H.17. 89L -do- Q7. 14A John Hall's Liquid Blue Envelope 8/949/233 -do- 8/949/252 John Hall's Transparent Amber 8/949/444	Docker's Stop Off Lacquer, Docker's 3:2 Stop Off - Strip Off Richardson's Adcora Hypalon H2. Croda Ltd. Rhinohide

3.3.7 Maskant evaluation in the 20:5 etchant

The specimens were hardened by martempering followed by descaling and degreasing as in Section 3.3.2.

The maskants evaluated are listed in Table IV. In most cases the pattern was cut in the maskant after the stoving procedure, the one exception to this being in the case of the Docker's 3:2 Stop-Off Strip-Off lacquer where the pattern was cut before stoving.

Table IV/

Table IV

Maskants Evaluated in the 20:5 Etchant

Neoprene Type	Vinyl Type	Miscellaneous
British Paints PR.785 -do- PR.790 Adcora C.E.R. 4(E)	Cellon's Birlon SL 6098 John Hall's Liq. Blue Envelope 8/949/252 John Hall's Liq. Green Envelope. Cellon's Birlon No.2 Clear	Docker's 3:2 Stop-Off Strip-Off

The vinyl and miscellaneous type lacquers were stoved for 30 min. at 120°C., while the neoprene type required 1 hour at 20°C. followed by 1 hour at 100°C.

It was generally found that line definition was poor, with the exception of the John Hall's Liquid Blue Envelope 8/949/252 lacquer which showed reasonable results. Double edging was evident on all specimens when the etch rate was below 0·100 in./hr. At higher etch rates there was an absence of double-edging, valleying and channelling, but undercutting was noticed and straight lines tended to become bowed (Fig.9).

Varying thicknesses of maskant gave no improvement in results and it was concluded that in this instance no maskant is completely suitable. The results obtained using the John Hall's Liquid Blue Envelope were the most encouraging. The acid resistance and ease of stripping of this lacquer were good.

3.3.8 Discussion

Although much work was carried out it was difficult to find a maskant which would satisfy all the requirements. Initial work, which indicated satisfactory results, could not be reproduced. Furthermore, batch variations gave unpredictable results, while different surface preparations gave similar inconclusive evidence.*

3.3.9 Conclusions

It is concluded that the most suitable maskant for the protection of 3% chromium - molybdenum - vanadium steel during chemical contouring is John Hall's Liquid Blue Envelope 8/949/252 at a thickness of 0·007 in.

The lacquer is stoved for 30 min. at 120°C, the pattern being cut after stoving.

4./

*Richardson, Ltd., ask the publishers to state that whilst Neoprene resists have not shown up as satisfactory maskants in this report, they are currently being employed successfully in the contour etching of aluminium and other alloys.

4. Etchants and Maskants for 5% Cr-Mo-V Steel Sheet (H.50)

4.1 Material

The material used, H.50 steel, was obtained from Jessop-Saville Ltd., in the form of 2 in. wide, 8 S.W.G. strip (Batch J.21).

Specimens cut from the strip were hardened by austenitizing 1,000°C. for 30 min. in an atmosphere of argon, quenched in Edgar Vaughan's 2E Oil, and tempered at 550°C. for 60 min.

Hardness tests were carried out on all specimens prior to contouring, the hardness range being 564-626 HV., equivalent to 121-134 tons/sq.in. tensile strength.

4.2 Development of Etchant for 5% Cr-Mo-V Steel Sheet

4.2.1 Surface pretreatment

Specimens were pickled in Ferroclene 100 and degreased in Collex 304 alkaline metal cleaner.

It has been indicated previously (Section 3.2.2) that rough surface condition of the initial material gives rise to a poor finish after contouring in some etchants, and that linishing of the rough surface is a pre-requisite to obtaining a good surface finish. Whereas this treatment may be carried out easily on small flat objects, it would be difficult on larger manufactured articles. With this in mind it was decided that surface improvement treatments would not be given to the specimens prior to etching. Any etchant failing to produce a smooth surface, therefore, was rejected.

4.2.2 Initial etchant development

Preliminary etching trials were carried out using the etchant previously developed for 1% chromium - Molybdenum steel. Results achieved with this etchant (18% sulphuric acid - 5% nitric acid in water) were unsatisfactory, the edge definition being poor and the etch rate being less than 0·050 in./hr., even at temperatures up to 82°C.

Freshly prepared solutions of the 12% nitric acid - 5% sulphuric acid - 5% phosphoric acid in water etchant tried for the 3% chromium steel gave poor edge definition although improved results were obtained when the iron concentration in the etchant increased. The etch rate, however, was low and did not approach the target rate of 0·060 in./hr. at etchant temperatures below 82°C.

In view of these results it was decided to evaluate nitric acid solutions and to determine the effect of sulphate and phosphate ions added initially as their sodium salts.

Results of etching trials with 10%, 15% and 20% nitric acid and of nitric acid solutions containing sodium phosphate and sodium sulphate at a temperature of 70°C. are shown in Table V.

The nitric acid etchants gave acceptable surface finish and edge definition, but a satisfactory etch rate was obtained only with the 20% nitric acid solution. Additions of sulphate ions resulted in a decrease in etch rate and a deterioration in surface finish, and phosphate ions produced an even more marked reduction in etch rate though the surface finish was not affected.

Table V/

Table V

Etch Rates with Nitric Acid Etchants at 70°C

Nitric Acid (%)	Sodium Sulphate (%)	Sodium Phosphate (%)	Etch Rate (in./hr)	Surface Finish
10	-	-	0·013	Acceptable
15	-	-	0·043	-do-
20	-	-	0·110	-do-
15	5	-	0·012	Poor
20	5	-	0·060	-do-
15	-	5	0·015	Acceptable
20	-	5	0·036	-do-

The investigation into the effect of the addition of sulphate and phosphate ions as sodium salts was discontinued and further trials were carried out with corresponding additions in the form of acids.

Results of these tests, given in Table VI show that the addition of sulphuric acid to the nitric acid etchant, effects a remarkable increase in etch rate but with a corresponding decrease in surface finish. Phosphoric acid additions reduce the etch rate but have no appreciable effect on surface finish.

Comparison of the etch rates given in Tables V and VI shows that at a given total acidity, etch rates are lower if anions are added as sodium salts than as acids, suggesting that sodium ions have a positive inhibiting effect.

Table VI

Etch Characteristics of Binary & Ternary Acid Etchants

Etchant Composition (%)			Etchant Temperature (°C)	Etch Rate (in./hr)	Surface Finish	Edge Definition
Nitric Acid	Sulphuric Acid	Phosphate Acid				
15	5	-	70	0·20	Poor	Moderate
15	5	-	54	0·15	Poor	Poor
20	5	-	70	0·30	Poor	Moderate
15	-	5	70	0·040	Moderate	Moderate
20	-	5	70	0·052	Acceptable	Moderate
15	5	5	70	0·043	Acceptable	Moderate
20	5	5	70	0·140	Moderate	Acceptable

4.2.3 Further etchant development

In view of the results of the preliminary trials further work was concentrated on three etchants, these being:-

20% nitric acid
20% nitric acid, 5% phosphoric acid
20% nitric acid, 5% phosphoric acid, 5% sulphuric acid

The natural etchant life of these solutions was determined. Samples of H.50 steel of known thickness and weight were contoured for periods of one hour starting with a fresh etchant, until the etch rate fell below 0·040 in./hr.

At this stage the etchant was regenerated by the addition of nitric acid in an attempt to restore the activity to that of the original solution and further contouring was carried out. (The amounts of acid addition made were below that for exact restoration of the etchant activity and this necessitated a second addition earlier than it should have been required.). Regeneration was repeated twice. At this point the useful life of the 20% nitric acid - 5% phosphoric acid etchant was completed, an orange peel effect (Fig.7) produced on the etched surface of the steel after the second regeneration becoming more pronounced as the iron content of the etchant increased above 52 gm./litre.

The edge definition and surface finish produced by the 20% nitric acid solution were acceptable even after the third regeneration stage, at an etch life of 52·2 gms./litre. (Figure 15). The ternary acid solution, although having a life of 92 gm.iron/litre, produced a worse edge definition than that resulting from the 20% nitric acid etchant, and the surface finish in the early stages was of a lower standard. (Figure 16). No evidence of channelling or undercutting was noted with either of these two etchants.

4.2.4 Discussion

Evaluation of nitric acid etchants and etchants based on nitric acid solutions with sulphate and phosphate ions added either by means of their acids or sodium salts, indicated two promising solutions, these being 20% nitric acid in water and 20% nitric acid - 5% sulphuric acid - 5% phosphoric acid in water.

The ternary acid solution has a greater etch rate and a longer natural life than the 20% nitric acid etchant but, particularly in the early stages, the surface finish and edge definition were inferior to those produced by the single acid solution.

No channelling or undercutting was noted with either of these etchants and both were suitable for contouring without pre-treatment of the steel surfaces, the etch action having a smoothing effect on initially rough surfaces.

4.2.5/

4.2.5 Conclusions

The preferred etchant for chemical contouring of 5% chromium - molybdenum - vanadium steel is 20% nitric acid in water. An etch rate of 0·10 in./hr. is achieved with a fresh solution at 70°C and the etchant life is up to at least 50 gm.iron/litre. Etchant regeneration can be carried out by the addition of nitric acid when the etch rate has fallen below a specified figure (0·04 in./hr. used in this investigation).

Surface pre-treatment is not necessary as initially rough surfaces are smoothed during contouring.

4.3 Evaluation of Maskants for 5% Cr-Mo-V Steel Sheet

4.3.1 Material

Tests were done on material from batch J.21. The composition and heat treatment have been described in Section 4.1.

4.3.2 Surface pretreatment

The specimens were acid pickled in Ferroclene 100 to remove the heat treatment scale, and immersed in 'Collex 304' alkali cleaner to give a clean lacquer-adherent surface. Previous experience had shown that the surface finish of the materials received for contouring could vary between rather wide limits, i.e. approx. 200 micro in. (rough) to approx. 60 micro in. (smooth). As the material used in the maskant selection trials had a rough "as received" finish, a linishing treatment was carried out on 50% of the samples to enable contouring tests on smooth, in addition to rough, specimens.

4.3.3 Maskant evaluation

The trials were carried out using the etchant finally chosen for this steel (Section 3.2) (20% nitric acid in water at 70°C.) and the maskants evaluated are detailed in Table VII, curing being carried out as specified by the manufacturers.

TABLE VII/

Table VII

Maskants Evaluated for 5% Chromium - Molybdenum - Vanadium Steel

Neoprene Type	Vinyl Type	Miscellaneous
Richardson's C.E.R.4(E)	Docker's Strip-Off Lacquer (Heavy Bodied Type)	Docker's Stop-off Lacquer
Docker's T.G.X. 69		-do- 3:2 Stop off Strip off
British Paints PR.790	John Hall's Liq.Blue Envelope 8/949/233	Croda Rhinohide Lacquer
-do- PR.787	-do- Liq.Blue Envelope 8/949/252	
-do- PR.785	-do- Transparent Amber 8/949/444	
	Cellon's Birlon No.3	
	-do- Q7/14A	
	-do- SL/6098	
	-do- H17/89L	

The results obtained indicated that the neoprene type maskants were unsatisfactory due to excessive scalloping and poor edge definition.

The results from the vinyl type lacquers were found to depend on the metal surface condition (Figs. 10 & 11). Examples of this were especially pronounced with Cellon's SL/6098, Q7/14A, H17/89L and John Hall's Transparent Amber 8/949/444, where rough surfaces gave acceptable edge definition and smooth surfaces poor edge definition, after chemical contouring.

Cellon's H17/89L, John Hall's Transparent Amber and Docker's Strip-off Lacquer were extremely difficult to strip from the specimens after contouring. Docker's Strip-off lacquer also suffered from a bleaching action and embrittlement on coming into contact with the etchant.

John Hall's Liquid Blue Envelopes 8/949/233 and 8/949/252 and Cellon's Birlon No. 3 proved satisfactory on both rough and smooth surfaces.

In the miscellaneous group, Docker's Stop-off lacquer gave satisfactory line definition but could not be stripped from the metal surfaces and Croda Ltd's Rhinohide lacquer proved similar to the Docker's Strip-off lacquer. Docker's 3:2 Stop-off Strip-off mixture was found to be satisfactory on both rough and smooth surfaces but giving better results on smooth surfaces.

At this point it was decided to continue more specific tests on the three most satisfactory maskarts, these being Docker's 3:2 Stop-off Strip-off mixture and John Hall's Liquid Blue Envelopes 8/949/252 and 8/949/233.

The/

The viscosity of the lacquers was standardized within the limits 35-40 sec., using a standard No. 4 Ford Cup Viscometer, and varying maskant thicknesses were tried, the best results being obtained with coatings of 0•007 - 0•009 in. These thicknesses showed good acid resistance and edge definition and were considered to be the most economical from both a time and material point of view. The best masking results were obtained on specimens coated by dipping into the lacquer.

For the Docker's mixture, the required pattern was scribed before the maskant was cured at 120°C. for 30 min. However, it was found that if the vinyl lacquers (John Hall's Liquid Blue Envelopes 8/949/252 and 8/949/233) were subjected to a similar treatment, the edges became ill-defined after curing at 120°C., due to the molten nature of these lacquers at this temperature. It was therefore desirable to scribe the pattern on the maskant after it had been cured.

4.3.4 Discussion

The selection of one maskant to cover the contouring of both rough and smooth surfaced specimens of H50 steel is not entirely satisfactory since a maskant fulfilling the requirements characterized by a smooth surface does not necessarily produce satisfactory results on a rough surface and vice versa.

The problem was especially pronounced with the vinyl type lacquers and may be related to the fact that at the stoving temperature the vinyl coating tends to become molten.

In these conditions the coating may flow into the pits of an uneven surface to give good adhesion, but a smooth surface will offer no favourable adhesive properties.

4.3.5 Conclusions

The most satisfactory results were exhibited by Docker's 3:2 Stop-off Strip-off mixture which had been cured at 120°C. for 30 min., the pattern having been scribed before curing.

If it is necessary to standardize on one material for both types of surface finish this lacquer is recommended. However, John Hall's Liquid Blue Envelope 8/949/252, the pattern being cut after curing at 120°C. for 30 min., produced very satisfactory results on a rough surface only.

In both instances a coating thickness 0•007 - 0•009 in. is recommended.

5./

5. General Etchant for Low Alloy Chromium Bearing Steels

5.1 Introduction

The preferred etchants developed so far are:

RS.130	1% Cr-Mo	18% sulphuric acid - 5% nitric acid at 70°C
RS.140	3% Cr-Mo-V	20% nitric acid - 5% phosphoric acid at 60° to 70°C
H.50	5% Cr-Mo-V	20% nitric acid at 70°C.

It was then decided to attempt to find a single etchant adequate for all three steels, as standardisation might be convenient and economic.

5.2 Materials

The following materials were used:

RS.130 Batch B/BU.10. Composition:- 0·33%C, 0·56%Mn, 0·31%Si, 0·006%S, 0·009%P, 0·10%Ni, 1·00%Cr, 0·16%Mo, rem. Fe. sheet 0·104 in. thick, from Burys and Co. Ltd. austenitised at 900° for 40 min. in air, quenched in G.2 oil and tempered at 450°C. for 60 min.

RS.140 Batches BU.514 and 677 austenitised and tempered as described in Section 3.1.

H.50 Batch J.21 austenitised and tempered as described in Section 4.1.

Specimens were pickled in Ferroclene 100 and degreased in Collex 304 alkaline metal cleaner and masked with the appropriate preferred lacquers (Sections 3.3.9 and 4.3.5).

5.3 Development of Common Etchant

5.3.1 Common etchant trials

During the etchant development programme for H.50 steel (Section 4.2.2) samples of RS.130 and RS.140 material were also contoured in the etchants under investigation. Smooth surfaces were produced on both the RS.130 and RS.140 specimens in the 20% nitric acid - water etchant at a temperature of 65°C. but for the RS.130 steel the etch rate was only 0·017 in./hr. Additions of sulphuric acid to the etchant increased the etch rate but a marked deterioration of surface finish was noticed.

Work was also carried out in the preferred etchant for RS.140 steel. Satisfactory results were obtained for H.50 steel, but the RS.130 steel etch rate was again exceptionally slow.

Consideration/

Consideration of the etchant developed for RS.130 steel suggested that high sulphuric acid content etchants might prove to be a satisfactory general etchant. Results proved rather unsatisfactory, with passivation occurring when the sulphuric acid content exceeded 20%. Additions of nitric acid to these types of etchant gave an increased etch rate but temperatures as high as 82°C. were required before any appreciable etching took place and the resulting surfaces of the specimens were very rough.

Because of these results it was decided to concentrate work upon a high nitric acid content etchant, namely the 20% nitric acid - 5% sulphuric acid - 5% phosphoric acid etchant which had already been tried for the H.50 steel (Section 4.2.3).

5.3.2 Etchant development

Results from previous work indicated that the different steel alloys would etch at different rates for any given temperature. This fact was confirmed and it was therefore necessary to determine the most suitable operating temperature of the etchant for each type of steel.

It was found that, using the 20% nitric acid - 5% sulphuric acid - 5% phosphoric acid etchant, the best results were obtained with RS.130 steel at 57°C. - 63°C., with RS.140 steel at 63°C. - 68°C. and with H.50 steel at 65°C - 70°C. These temperature ranges were strictly adhered to throughout the etchant evaluation. (Figures 17, 18 and 16).

The life of the 20:5:5 etchant varied from alloy to alloy, but even at the end of its life the etch rate was as high as 0·060 in./hr. for RS.130 and RS.140 steels. Although the etch rate at the end of the etchant life was slower for H.50 steel, the maximum iron content tolerable was high for this material. Regeneration, in order to restore the etchant to its original condition, was effected by the addition of 1 litre of concentrated nitric acid to every 20 litres of etchant. The H.50 steel was etched in a satisfactory manner by the regenerated etchant, but the RS.130 and RS.140 steels exhibited a marked deterioration in the quality of the surface finish. Overall, however, there was the predicted increase in etch rate for all the alloys and as the etchant life before regeneration was high the etchant was considered to be suitable for general use.

It is interesting to note that as the dissolved iron content of the etchant increased, the surface condition of the H.50 material improved. This was not the case with the RS.130 and RS.140 alloys. (Figures 17, 18 and 16).

5.3.3 Discussion

An etchant that was investigated during trials with H.50 steel, namely 20% nitric acid - 5% sulphuric acid - 5% phosphoric acid, has been found to be suitable for general use provided specified temperature limits are adhered to.

The/

The temperature of the etchant is very important, since if it becomes too high while contouring RS.130 and RS.140 steels, the etch rate rises to at least 0·200 in./hr. which is an unacceptable rate for close tolerance work. A temperature below the specified limits for the RS.130 and RS.140 steels leads to a rough surface finish on the specimens and channelling becomes evident.

In the case of the H.50 material, an excessive temperature gives rise to a deterioration of surface finish and a lower than specified temperature slows the etch reaction to an uneconomical rate.

Because of difficulties in analysis of a ternary acid bath, it is uncertain what acid addition is necessary to return the etchant to its original condition. However, the etchant life may be extended by periodically adding 20% nitric acid solution, sufficient to maintain the original acid content of the bath. It has been found that this type of regeneration, when etching RS.130 and RS.140 steels, ultimately gives rise to an uneven but smooth surface finish on the specimen (Figure 8) and striations, both conditions being undesirable.

Regeneration of the etchant while contouring H.50 steel gave an excellent surface finish and good edge definition up to at least 92 gms/litre of dissolved iron.

5.3.4 Conclusion

Development work has shown that none of the etchants selected for the individual alloys is suitable to be classed as a universal etchant.

The most satisfactory general results were obtained using the 20% nitric acid - 5% phosphoric acid - 5% sulphuric acid mixture within the following specified temperature limits:

RS.130 - 57°C. - 63°C.

RS.140 - 63°C. - 68°C.

H.50 - 65°C. - 70°C.

6. Preparation of Contoured Test Panels

6.1 Introduction

In order to demonstrate the capabilities of the chemical contouring processes, demonstration panels of each steel, RS.140 and H.50, were prepared and etched to the dimensions given in Figure 19.

6.2 Materials

The materials were obtained in the form of panels which had been guillotined from "as-received" sheet.

The/

The RS.140 material was from Batch BU.514 (Table I) in the form 12 in. x 8 in. x 0·170 in. The H.50 steel was from Batch BU.227 in the form 12 in. x 8 in. x 0·098 in., and one panel was obtained from an unknown batch in the form 12 in. x 8 in. x 0·150 in.

The chemical composition of the new H.50 material is given in Table VIII.

Table VIII

Material	Chemical Composition (%)								
	C	Mn	Si	S	P	Ni	Cr	Mo	V
Batch BU.227	0·41	0·50	0·90	0·012	0·009	0·29	5·08	1·35	1·11
H.50 Specification Limits	0·35/ 0·40	0·40/ 0·50	0·95/ 1·15	0·03 max	0·03 max	- -	4·8/ 5·2	1·25/ 1·45	1·0/ 1·2

The panels were heat treated as before (Sections 3.1 and 4.1), pickled in Ferroclene 100 and degreased.

6.3 Masking

The maskant used for the protection of the RS.140 material was John Hall's Liquid Blue Envelope 8/949/252 and for H.50 a mixture of Docker's Stop-off Strip-off lacquer in the proportion of 3:2 by volume.

Both types of lacquer were standardised to produce a viscosity of 35-40 sec., using a Ford No. 4 Cup Viscometer, by the addition of appropriate thinners.

The panels were given a series of sprayed coatings until the maskant thickness approximated to 0·010 in. The edges and corners of the panels, as in all the previous contouring work, were further protected by silicone adhesive polyester tape. The tape was applied after stoving the maskant material under the recommended condition.

A template containing the required geometrical forms was placed on each panel in turn and secured with adhesive tape. The patterns were then cut in the maskant using a surgical scalpel. The template was removed and the maskant stripped from the areas to be etched.

The pattern was scribed on the maskant of the RS.140 material after stoving, but before stoving in the case of the H.50 maskant.

The template had previously been cut from a fibre glass laminate approximately 0·085 in. thick, the pattern having been cut to allow for eat-back under the maskant during contouring.

The/

The term "eat-back" means the extent to which etching occurs along the specimen surface underneath the maskant. The eat-back should, theoretically, equal the etch depth, but in practice it varies between 80% and 100% of the etch depth. The etch depth specified for the test panels was 0·12 in. and the template was cut to allow for an eat-back of 0·12 in. Four of the five H.50 panels were, however, too thin to allow an etch depth greater than 0·085 in., but for convenience the single template was used.

6.4 Etching process

In each case the panel was placed in the contouring bath in a horizontal plane with the face to be etched uppermost.

The contouring of the RS.140 material was carried out in a stainless steel tank fitted with two electrical immersion heaters. The etchant composition was 20% nitric acid, 5% phosphoric acid in water at 60°C. The etch rate was approximately 0·085 in./hr.

The H.50 steel was contoured in a polythene tank containing a 20% solution of nitric acid at 65°C., the etchant again being heated by immersion heaters. The etch rate was approximately 0·090 in./hour for all the panels.

6.5 Results of RS.140 panels

Four panels of RS.140 steel were contoured. The surface finish achieved was good and though there was some evidence of double edging and scalloping, the edge definition was acceptable. All panels showed a considerable variation in etch depth, the final thickness being less in the centre of the etched areas than at the edges. This difference in etch depth was greater than expected and no explanation was apparent. Final thickness measurements on the panels are detailed in Figs. 20 to 23 and panel No. 1 is shown in Fig. 24.

6.6 Results of H.50 panels

The contouring of four H.50 panels posed a problem because of their thinness. The drawing stipulated at etch depth of 0·120 in. but as the panel thickness was 0·098 in. an etch depth of 0·085 in. was selected. Sufficient material of 0·15 in. thickness was found to provide a further panel (No. 5) but no information regarding this material was available.

While attempting to obtain the best overall results, two of the five panels showed eat-through due to the variation in etch depth. Satisfactory results were obtained with the remaining three panels. Final thickness measurements on the three satisfactory panels are given in Figs. 25 to 27 and panel No. 1 is shown in Fig. 28.

6.7 Discussion

Difficulty was experienced in obtaining satisfactory line definition results from the contouring of the RS.140 steel panels. Earlier work indicated that the maskant selected for the work suffered limitations and the indifferent results were not wholly unexpected. All the RS.140 panels showed a considerable variation in etch depth.

The/

The H.50 material showed satisfactory results with respect to line definition and surface smoothness. The etch depth variation was not as great as that noted with the RS.140 panels. The maskant suffered a bleaching action during contouring but this fact seemed to have no deleterious effect on the results.

For both materials, the etch depth adjacent to the edges was, in general, less than in the centre of the etched pockets. This etch depth variation was greater than expected and no satisfactory explanation has been formulated.

6.8 Conclusions

The four RS.140 steel panels were satisfactory as regards surface finish but they all showed a variable etch depth. Considering all four panels, this variation was from 0·106 in. to 0·138 in. in the centres of etched pockets and 0·094 in. to 0·128 in. towards the edges of the pockets.

Two of the five H.50 steel panels were eaten through in places due to a variable etch rate but the remaining three panels were satisfactory. On the two panels initially 0·098 in. thick, etch depth varied from 0·078 in. to 0·090 in. in the centres of the etched pockets and from 0·073 in. to 0·088 in. towards the pocket edges.

7. Sustained Load and Fatigue Tests

The effect of contouring on the mechanical properties of steels has been studied, particular consideration being given to the possibility of embrittlement due to absorption of hydrogen.

Sustained load tests have been completed on chemically contoured notched specimens of both steels. Reverse bend fatigue tests on contoured sheet specimens of both steels have also been completed.

Full details of these tests are given in D.Mat. & S. Report No. 115. No hydrogen embrittlement effects were found, but some reduction in fatigue properties was observed compared with mechanical machining.

8. Economics of Chemical Contouring

Many factors influence the economics of the process, hence an exact idea of costs cannot be determined.

Chemical contouring can only supersede machining when complicated forms and shapes are required or where the tensile strength of the material is so great that conventional cutting tools are of no use.

The cost of plant installation will depend on the size, shape and number of articles to be contoured, and the type of tank chosen. Ancillary equipment such as heaters, pumps, fume extraction, acid mixing facilities and lifting gear will also be required, also equipment for applying and stoving the maskant.

Etchant acids could be purchased in bulk, but storage facilities would be required. It is unlikely that maskant need be purchased in drums larger than 50 gallon capacity.

Assuming/

Assuming a reasonably large scale application of chemical contouring, the problem of etchant disposal would arise and unless the acid turnover exceeded about 100 gallons a day (which seems very unlikely) the acid could be either stored and carried away in a road tanker or disposed of, after neutralization, through the normal drainage system. The latter method would prove rather expensive.

The maximum labour cost would be incurred during the masking of the material, the handling of the specimen into and out of the etchant tanks and stripping of the maskant.

Table IX

Approximate Cost of Technical Acids

ACID		PRICE PER TON Carriage Paid	
		1 ton lots	1-5 cwt. lots
Sulphuric	(a) SG. 168°Tw. (R.O.V. Commercial in 10 gallon Carboys each 1 cwt. 2 qrs. 16 lbs. net.	£ 17 - 2 - 6	£ 26 - 17 - 6
	(b) SG. 140°Tw. (B.O.V.)	£ 14 - 2 - 6	£ 24 - 7 - 6
Nitric	S.G. 1.42 (70%) in approx. 10 gallon Carboys, each 1 cwt. 1 qr. net.	£ 33 - 10 - 0	£ 44 - 15 - 0
Phosphoric	S.G. 1.75 (88%) in approx. 10 gallon drums each 1 cwt. 2 qr. 7 lbs. net.	£149 - 10 - 0	£177 - 10 - 0

9. Discussion

Ideal results from chemical contouring would be perfect line definition, smooth etched surface and the complete absence of any other irregularities.

These results depend on three factors - the material to be contoured, the maskant, and the etchant and, on account of the many variables in each factor, an ideal solution to the chemical contouring problem is difficult to attain. For any given material a maskant and an etchant as a combination may give satisfactory results, but further complications arise as the results also depend on the actual composition of the batch of material and vary from batch to batch.

The surface condition of the material does not seem to influence the results except in the case of the H.50 steel, where maskant adhesion to a smooth surface is a problem. However, a satisfactory compromise can be reached by using the Docker's Stop-off Strip-off mixture.

Many/

Many different makes and types of maskant lacquer were used during the development programme. Generally speaking, the maskants normally displayed excellent stability during etching up to temperatures as high as 75°C. in the acids used. The ease of stripping of the maskant was, more often than not, good but the contouring results were often disappointing.

The number of etchants tried was more limited than the number of maskants mainly because the system of development adopted was to try the maximum number of maskants in a given etchant. The temperature of the etchant was usually kept at about 65°C. (± 50°C. for different etchants) any temperature above the stipulated one giving an appreciable rise in etch rate. The ideal etch rate was considered to be 0·060 in./hr. at which rate the contouring was controllable and economical.

The masking of the development specimens was carried out by a dipping process but the final test panels were spray coated. Since the most efficient and economical method of masking for production articles would be by spraying, the results obtained from the test panels are, in fact, the more realistic. It seems, however, that there is little, if any, difference between a contoured specimen which has been dip coated and one which has been spray coated.

The chemical contouring of RS.140 high strength steel can be accomplished to reasonable standards of line definition and surface finish of the etched areas. Slight double edging and scalloping were evident on the test panels, and etch depth over an etched area showed appreciable variation. The preferred etchant consists of a mixture of 20% nitric acid and 5% phosphoric acid in water. One major problem which arises with the use of this etchant is that the temperature has to be raised as the iron concentration increases. This in itself is a disadvantage and it must be concluded that contouring of RS.140 steel has not been accomplished completely satisfactorily. The best maskant found for use in conjunction with this etchant was John Hall's liquid Blue envelope.

The H.50 steel did not present such a complex problem as the RS.140 steel. The surface finish produced by etching was not unduly affected by the prior surface condition and the etchant bath was operated at constant temperature. Initial surface finish, however, affected maskant adhesion and resultant edge definition. The heat results on a rough surface were given by John Hall's liquid blue envelope, but Docker's 3:2 stop-off:strip-off mixture can be used for any type of initial surface finish. The preferred etchant is 20% nitric acid solution at 65°C.

A general etchant for RS.130, RS.140 and H.50 steels was difficult to find. The most encouraging results were observed using the 20% nitric acid - 5% phosphoric acid - 5% sulphuric acid within certain specified temperature limits (see para. 3.3.7).

Since the edges of the specimens to be contoured are often relatively sharp, polyester silicone tape should be used at all times to reinforce what could be a vulnerable part of the thin maskant film.

10. Conclusions

10.1 The preferred etchant for chemically contouring 3% Cr-Mo-V steel sheet is an aqueous solution of 20% nitric acid and 5% phosphoric acid, by volume of concentrated acids. The temperature of the etchant

must/

must be raised as iron accumulates in solution, i.e. 60°C. from fresh to an iron concentration of 12 g/l, then 65°C. to 20 g/l, then 70°C. to 30 g/l and regeneration.

Good surface finish and line definition were achieved and undercutting was negligible. The etch rate lay between 0·050 and 0·070 in./hr., but varied from one batch of sheet to another.

10.2 The preferred etchant for chemical contouring of 5% Cr-Mo-V steel sheet is 20% nitric acid in water at 70°C. An etch rate of 0·10 in./hr. is achieved with a fresh solution and the etchant life is up to at least 50 gm.iron/litre. Etchant regeneration can be carried out by the addition of nitric acid when the etch rate has fallen below a specified figure (0·04 in./hr. used in this investigation).

10.3 Development work has shown that none of the etchants selected for the individual alloys is suitable to be classed as a universal etchant. The most satisfactory general results were obtained using the 20% nitric acid - 5% phosphoric acid - 5% sulphuric acid mixture within specified temperature limits, which are:-

RS.130: 57°C. - 63°C.
RS.140: 63°C. - 68°C. and
H.50 : 65°C. - 70°C.

10.4 At a given total acidity, etch rates are lower if anions are added as sodium salts than if added as acids.

10.5 The most suitable maskant found for the protection of 3% Cr-Mo-V steel during chemical contouring is John Hall's Liquid Blue Envelope 8/949/252. The lacquer is stoved for 30 min. at 120°C., the pattern being cut after stoving.

10.6 For the protection of H.50 5% Cr-Mo-V steel during contouring the most suitable material is Docker's 3:2 Stop-off Strip-off mixture which has been stoved at 120°C. for 30 mins.

If it is necessary to standardize on one material for both types of surface finish this lacquer is recommended. John Hall's Liquid Blue Envelope 8/949/252, the pattern being cut after curing at 120°C. for 30 min., produced very satisfactory results on a rough surface only.

In both instances a coating thickness of 0·007-0·009 in. is recommended.

10.7 The four RS.140 steel panels were satisfactory as regards surface finish but they all showed a variable etch depth. The variation was of the order of ± 19%.

The H.50 panels showed similar satisfactory results and did not show such a variation of etch depth. The variation was of the order of ± 10%.

10.8 Chemical contouring has no effect on the notched sustained load properties of RS.140 steel at tensile strength levels of 105 and 115 tons/sq.in. and H.50 steel at 120 tons/sq.in. tensile strength.

11. Further Developments

Work is in hand to develop chemical contouring processes for two further types of steel sheet, (i) the 18% nickel maraging steel, and (ii) the R.A.R.D.E. copper-silicon steel aus-rolled.

Appendix I/

Appendix I

Definitions of terms used in Chemical Contouring of High Strength Steels

(i) The Strength and Quality of Acids used in the Formulation of Etchants

Nitric Acid	Technical quality	1·42 S.G.	70% W/W
Sulphuric Acid	Technical quality	1·84 S.G.	98% W/W
Phosphoric Acid	Laboratory Reagent quality	1·75 S.G.	88% W/W

(ii) Mean Etch Depth

The mean etch depth, more commonly termed the etch depth, on the standard 3in. x 3in. or 3in. x 1½in. test panels is obtained by locating eight points on the area to be etched.

The initial thickness of the material at these points is determined using a micrometer. After contouring the maskant is stripped and the points relocated so that a new series of readings is obtained.

(iii) Mean Etch Rate (in./hr.)

The mean etch rate is equivalent to the mean etch depth after contouring for one hour.

(iv) Surface Finish

The C.L.A. (centre line average) measurements obtained with a Talysurf instrument indicate the smoothness of a specimen. Surface finish does not indicate the degree of flatness which is generally assessed visually.

(v) Etchant Life

This term is defined as the maximum amount of iron that can be dissolved in the etchant without bringing about any appreciable deterioration in the surface finish and edge definition of the etched specimen. The etchant normally has at least one regeneration during its life. The units are gm. of iron dissolved per litre of etchant.

(vi) The Natural Etchant Life

Is taken to be the life of the etchant prior to the first regeneration.

(vii) Regeneration

Regeneration of an etchant involves the addition of fresh acid or a mixture of acids to an etchant which has become exhausted. The object of regeneration is to restore the etchant to its original character.

(viii)/

(viii) Passivation

Passivation is inertness of a metal surface to acid attack due to the formation of a surface film. Acid attack can be restored by removal of the film.

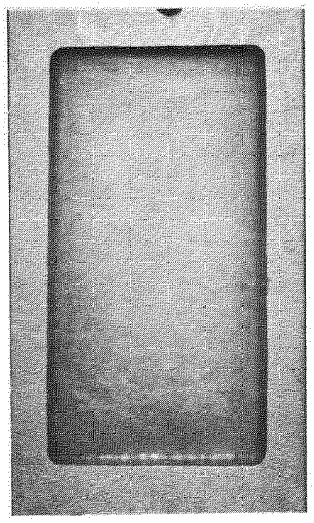


FIG. 1. GOOD LINE DEFINITION AND SURFACE FINISH

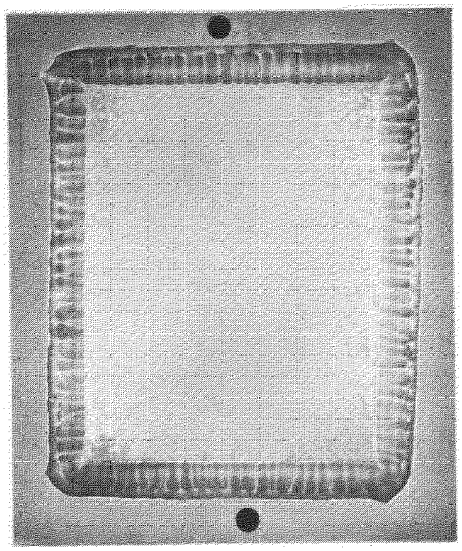


FIG. 2. CHANNELLING AND VALLEYING

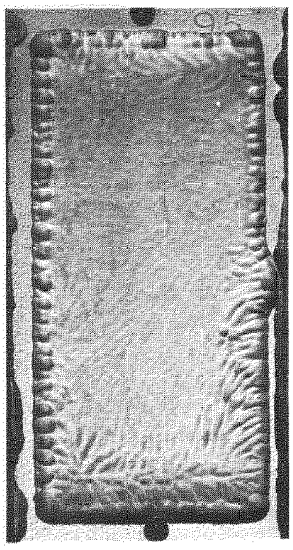


FIG. 3. STRIATIONS

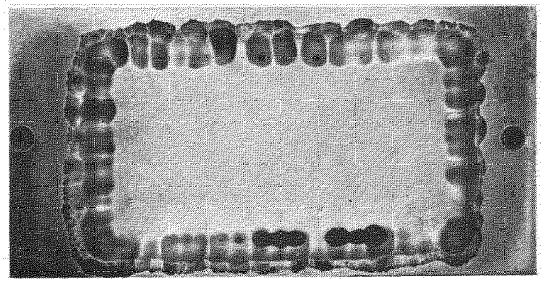


FIG. 4. SCALLOPING (MORE ADVANCED FORM OF VALLEYING)

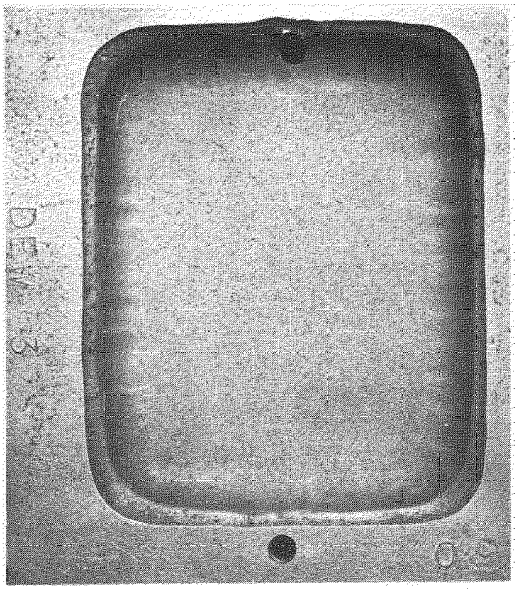


FIG. 5. DOUBLE EDGING

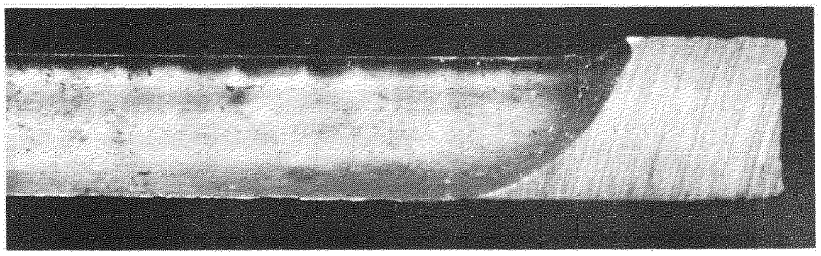


FIG. 6. UNDERCUTTING (X 6)

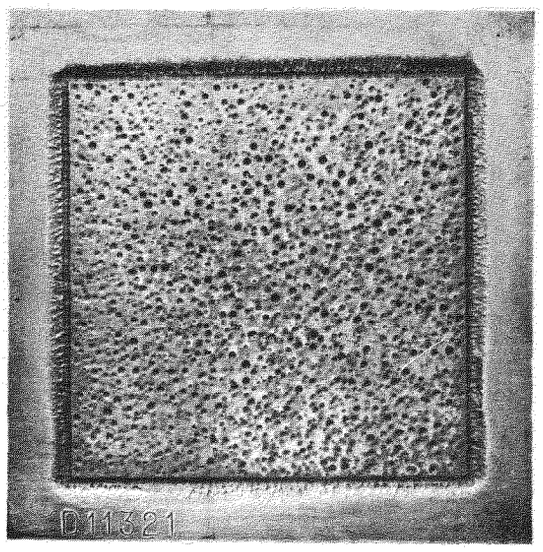


FIG. 7. ORANGE PEEL EFFECT

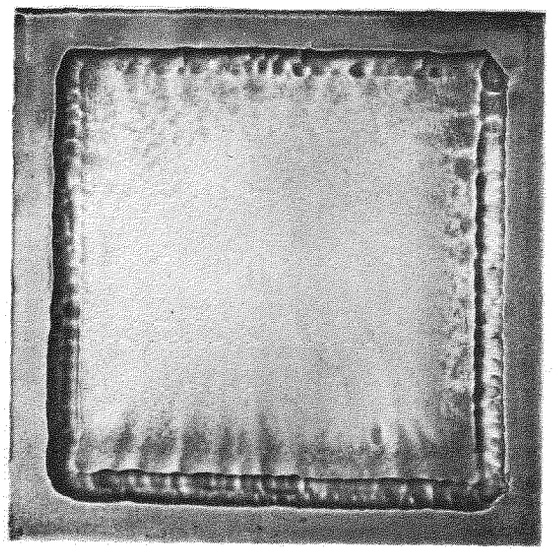


FIG. 8. SMOOTH BUT UNDULATING SURFACE

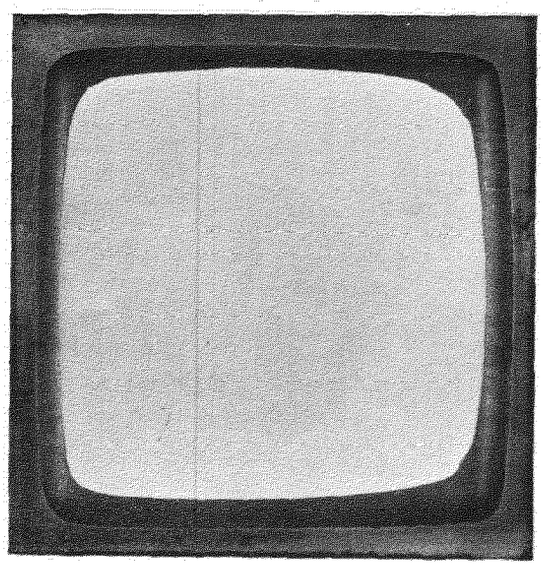


FIG. 9. BOWING

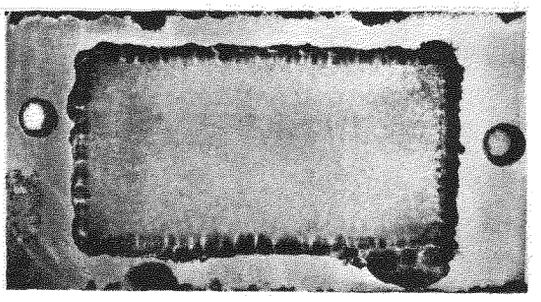


FIG. 10. EFFECT OF MASKANT ON LINISHED SURFACE

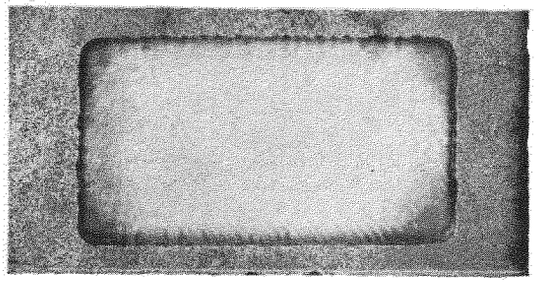


FIG. 11. EFFECT OF MASKANT ON ROUGH SURFACE

FIG. 12.

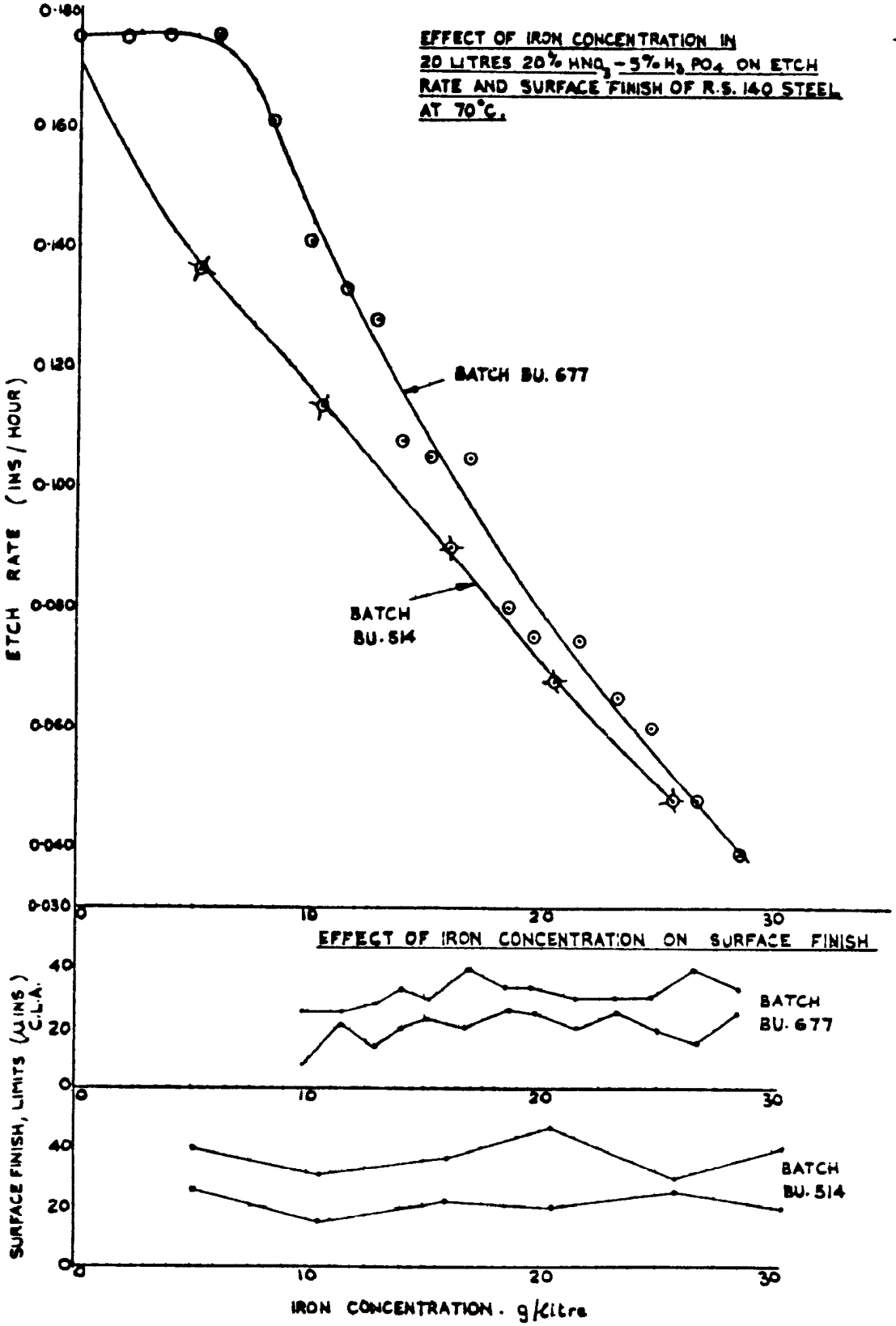
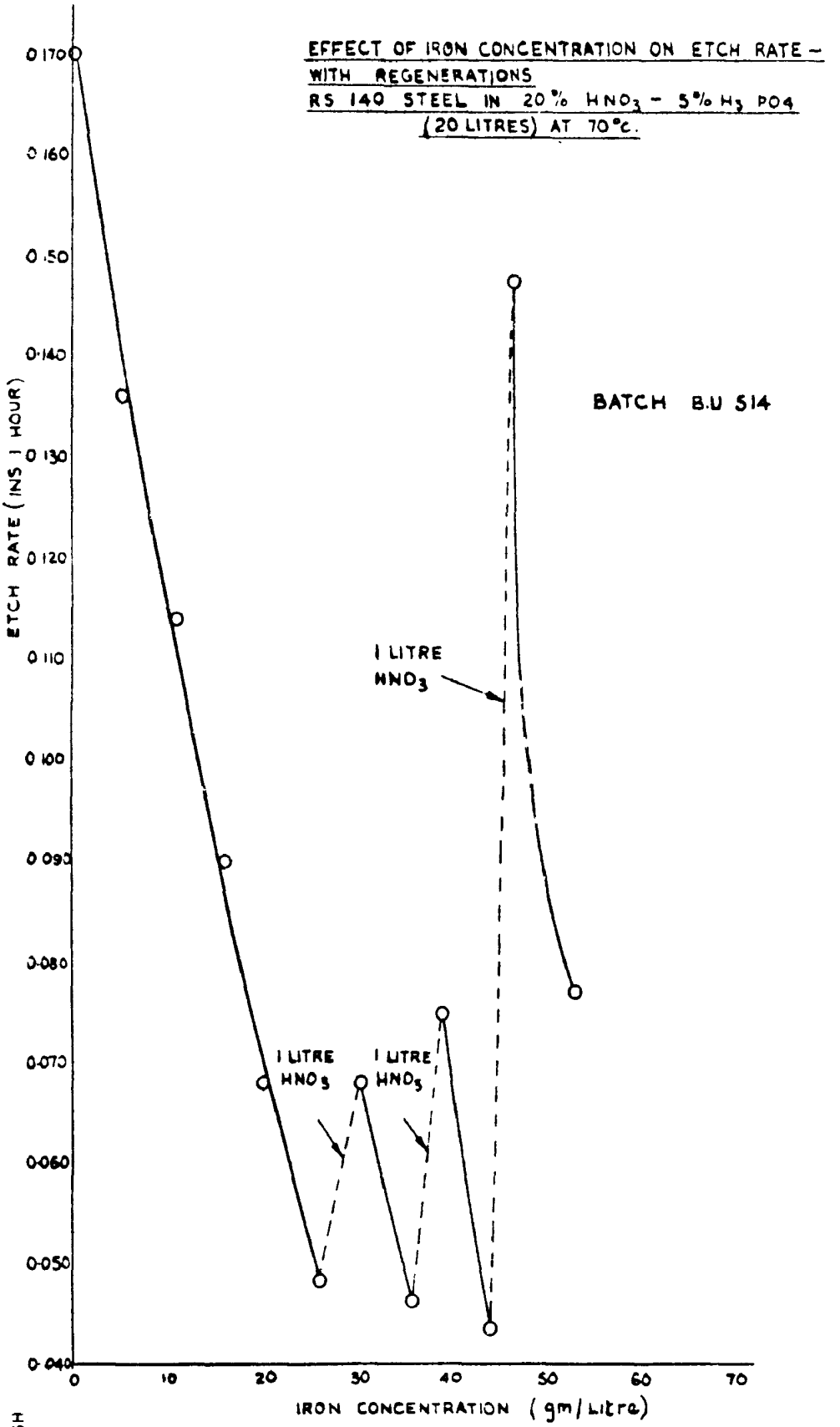
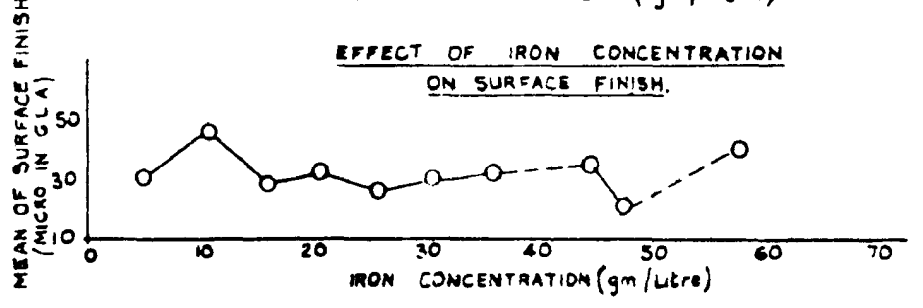


FIG. 12. BEHAVIOUR OF 3% Cr. STEEL IN 20% NITRIC ACID - 5% PHOSPHORIC ACID ETCHANT.

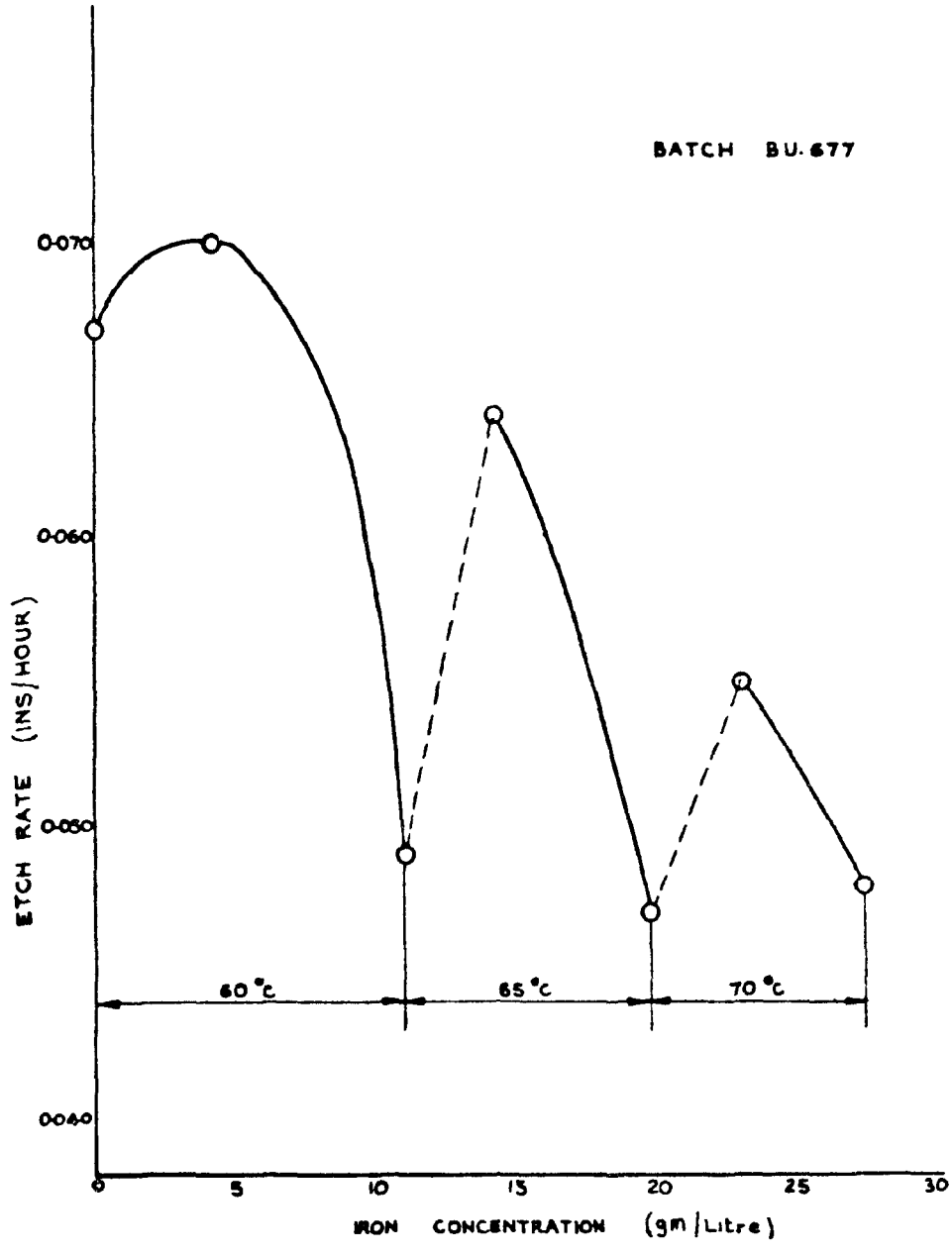
FIG 13





SA 4523

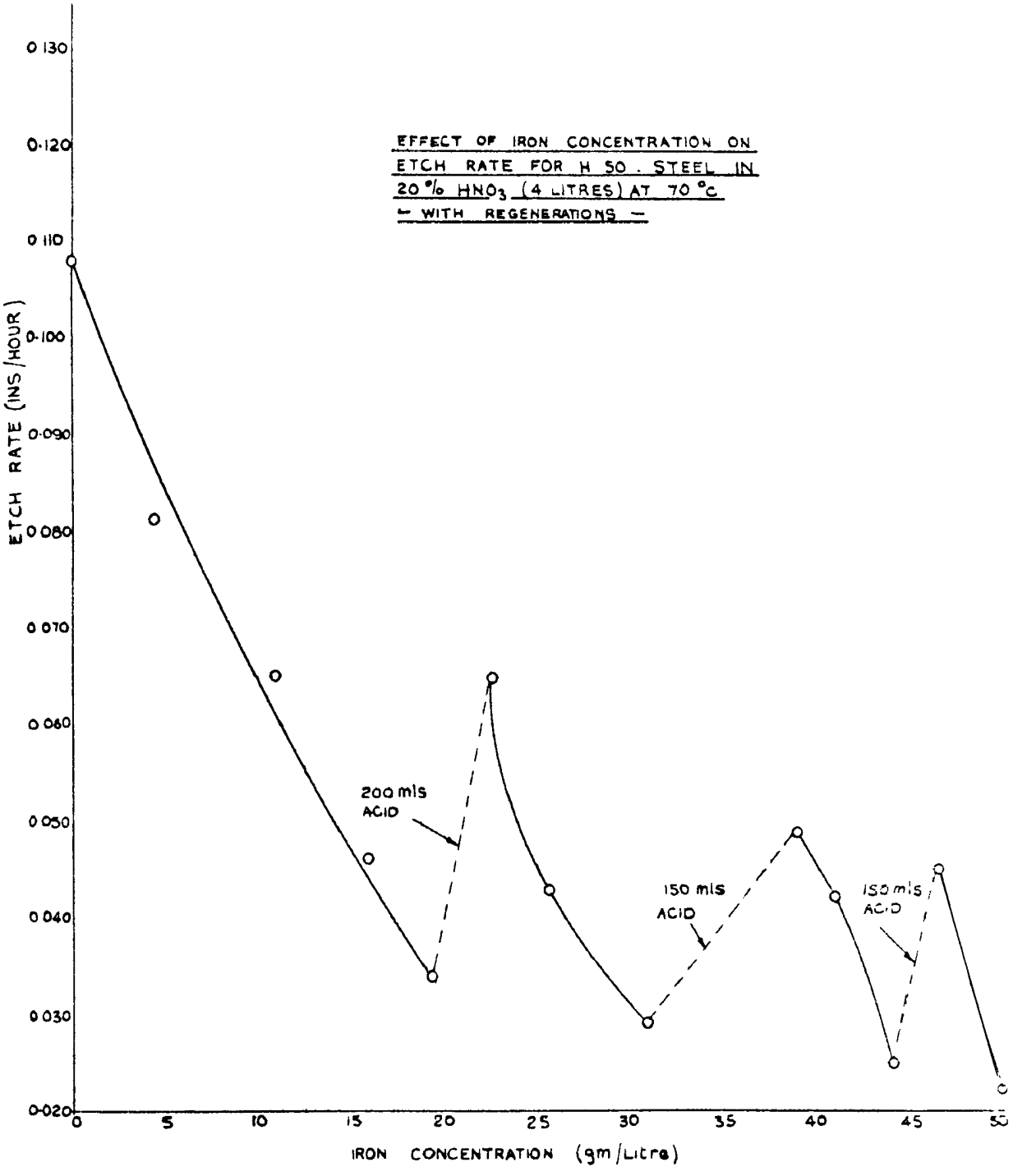
EFFECT OF IRON CONCENTRATION ON
ETCH RATE AT VARYING TEMPERATURES
RS.140 STEEL IN 20% HNO_3 – 5% H_3PO_4 (5 LITRES)



SA 4524

FIG. 14 BEHAVIOUR OF 3% Cr. STEEL IN 20% NITRIC ACID 5% PHOSPHORIC ACID ETCHANT

FIG 15.



EFFECT OF IRON CONCENTRATION ON SURFACE FINISH

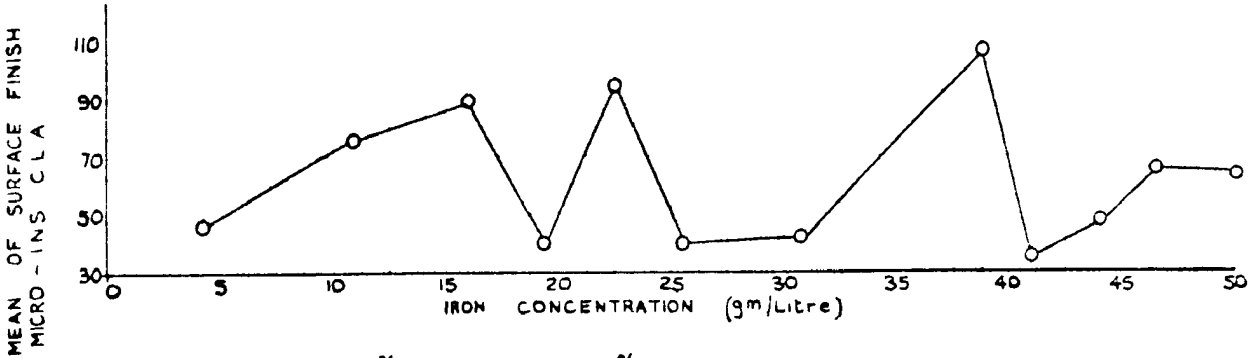


FIG. 15. BEHAVIOUR OF 5% Cr. STEEL IN 20% NITRIC ACID ETCHANT

SA 4525

FIG. 16.

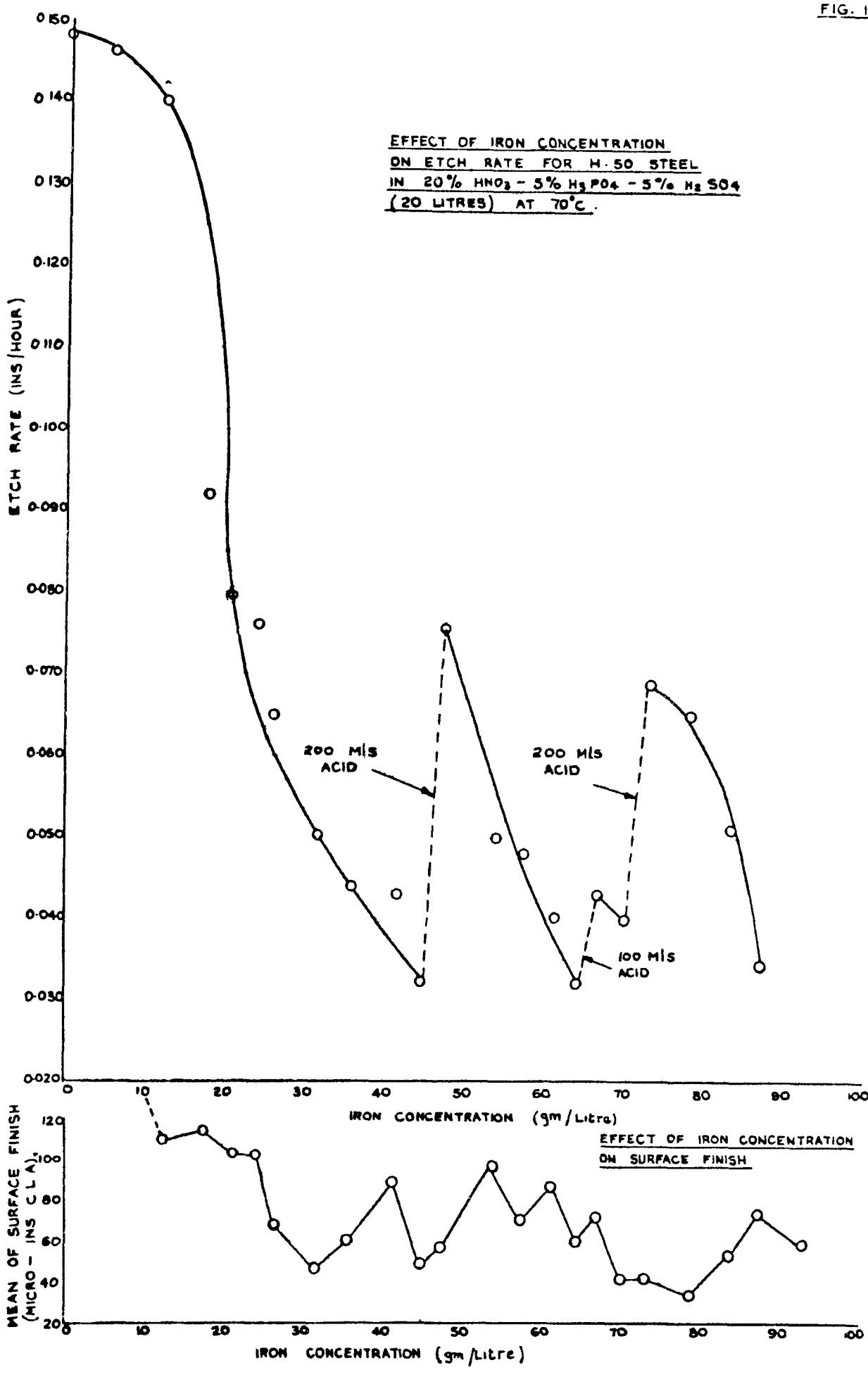


FIG 16. BEHAVIOUR OF 5% Cr. STEEL IN TERNARY ETCHANT.

SA 4526

FIG-17

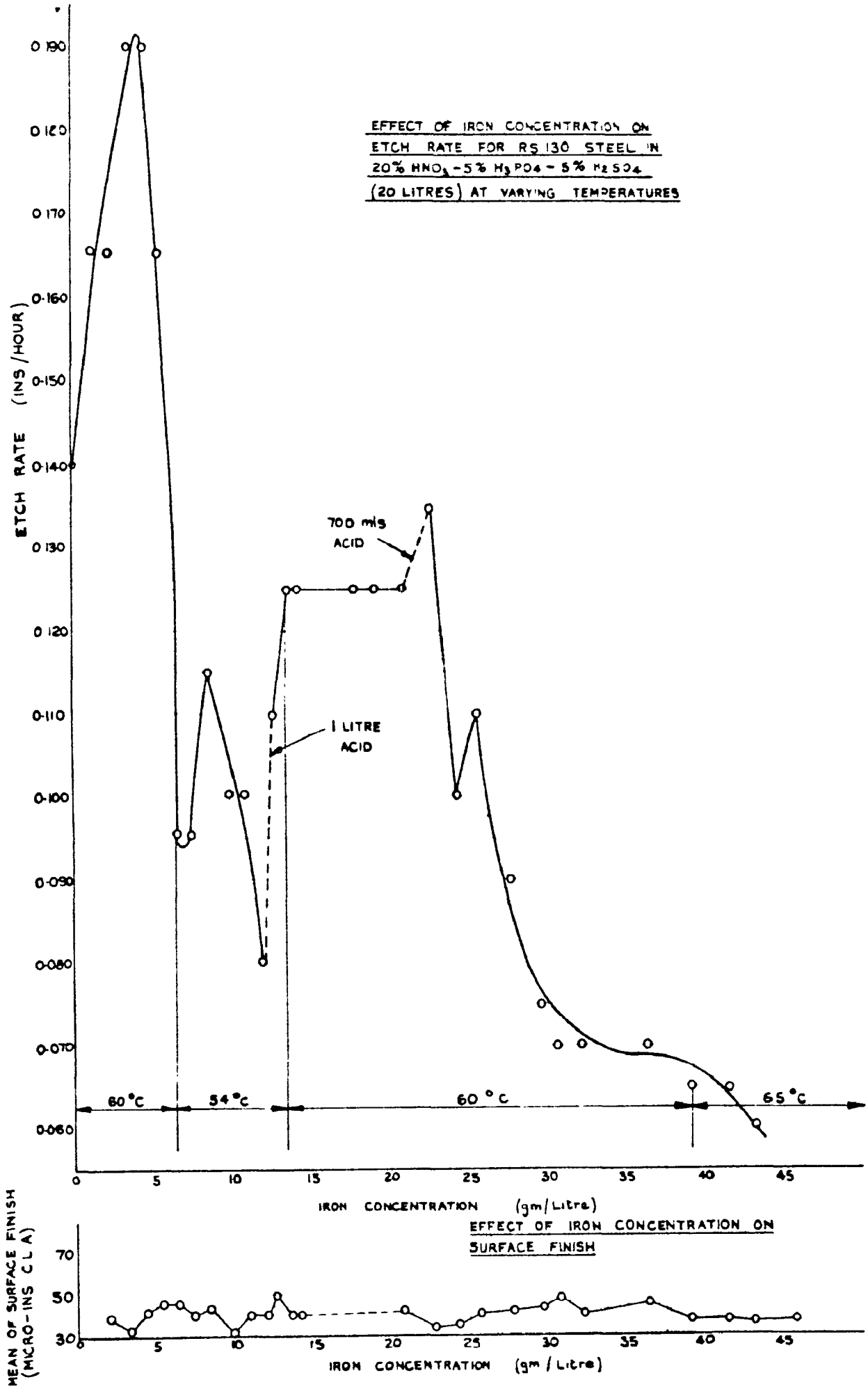
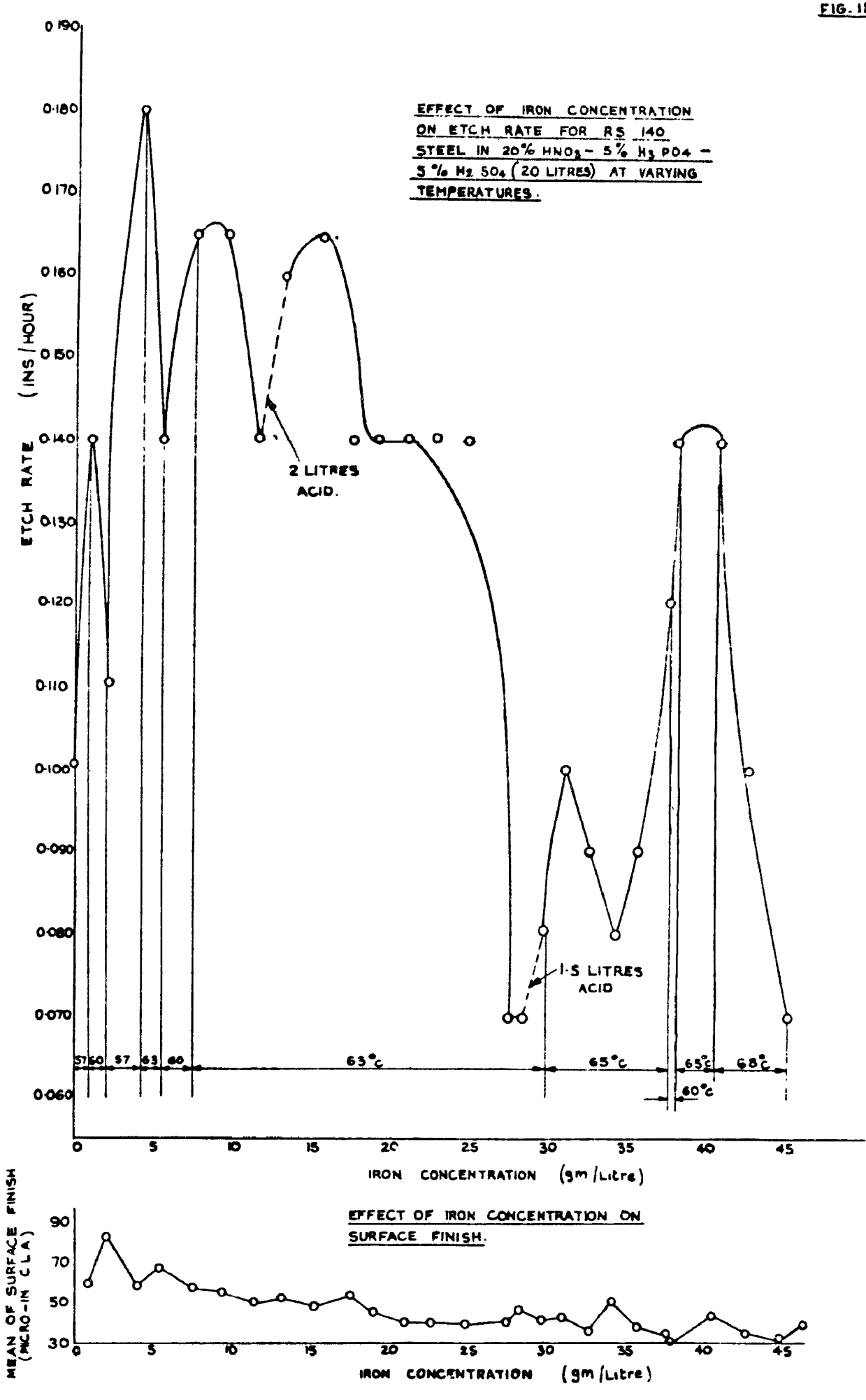
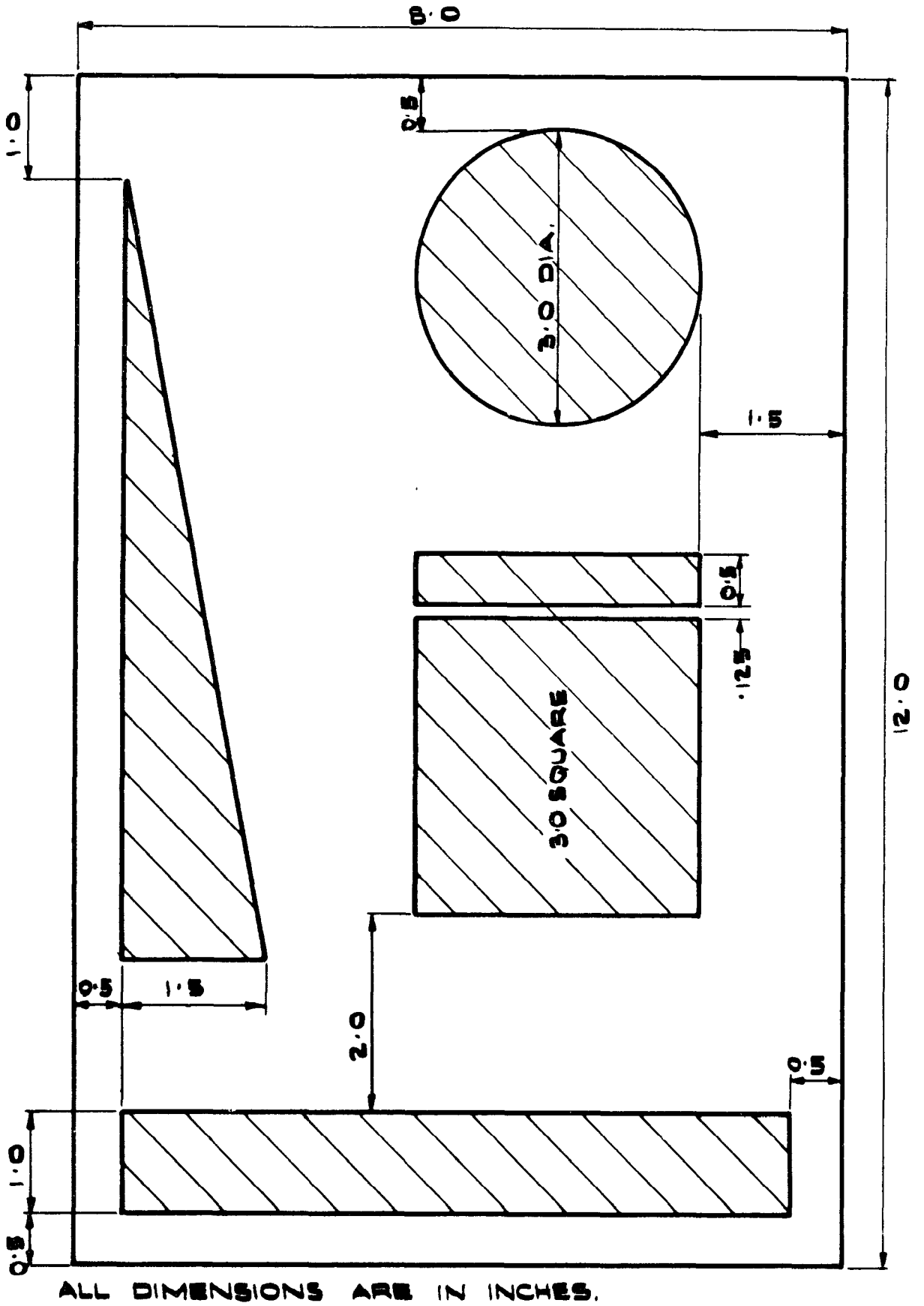

EFFECT OF IRON CONCENTRATION ON ETCH RATE FOR RS 130 STEEL IN 20% HNO3 - 5% H3PO4 - 5% H2SO4 (20 LITRES) AT VARYING TEMPERATURES
ETCH RATE (INS/HOUR)
0·190
0·180
0·170
0·160
0·150
0·140
0·130
0·120
0·110
0·100
0·090
0·080
0·070
0·060
700 mls ACID
1 LITRE ACID
60 °C
54 °C
60 °C
65 °C
0
5
10
15
20
25
30
35
40
45
IRON CONCENTRATION (gm/Litre)
EFFECT OF IRON CONCENTRATION ON SURFACE FINISH
MEAN OF SURFACE FINISH (MICRO-INS C.L.A)
70
50
30
IRON CONCENTRATION (gm/Litre)

FIG. 18.



SA 4528

FIG. 18. BEHAVIOUR OF 3% Cr. STEEL IN TERNARY ETCHANT.

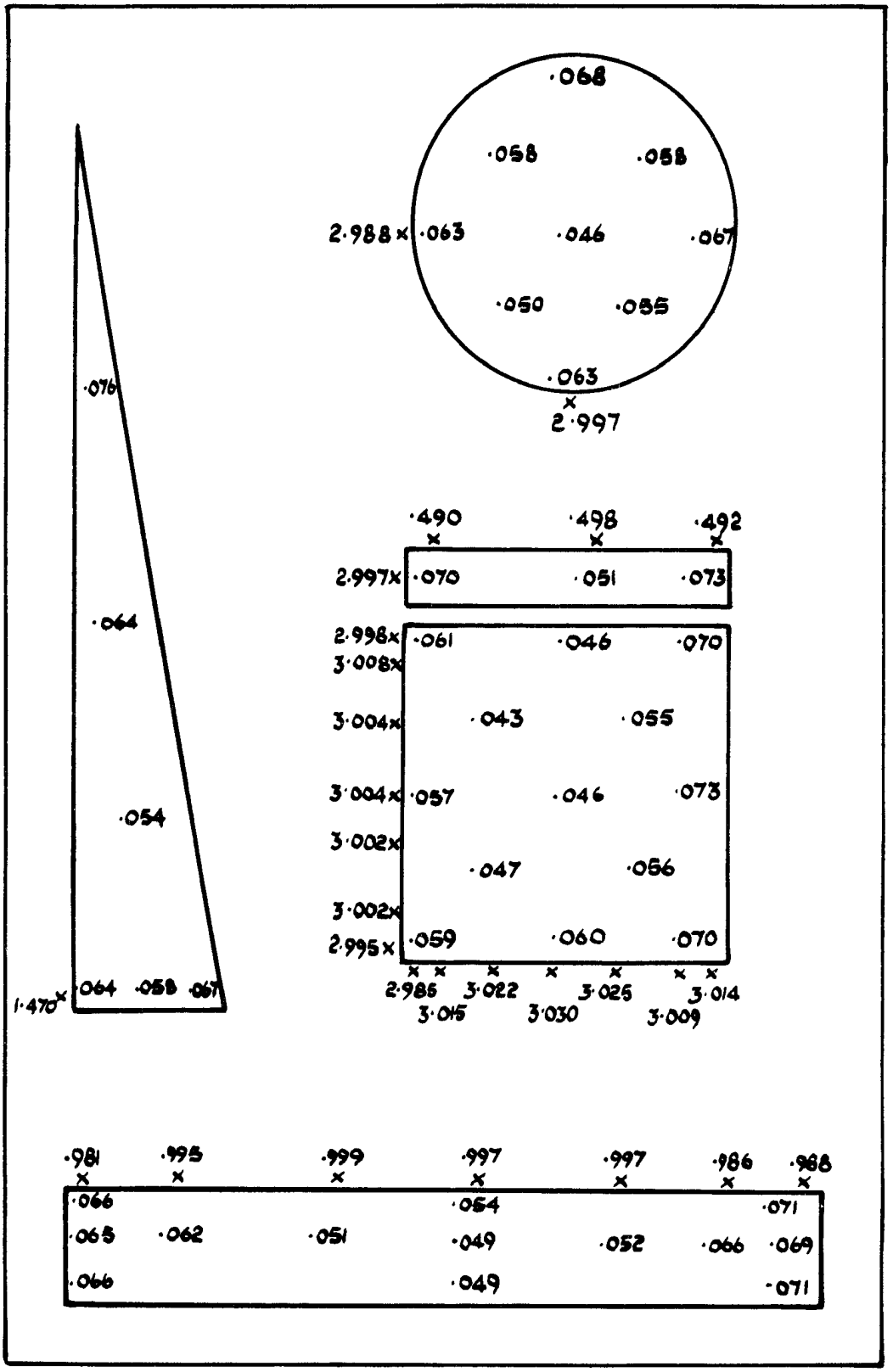


ALL DIMENSIONS ARE IN INCHES.

SHADED AREAS TO BE ETCHED TO GIVE FINAL DIMENSIONS AS SHOWN.

DRAWING OF TEST PANEL

FIG. 19

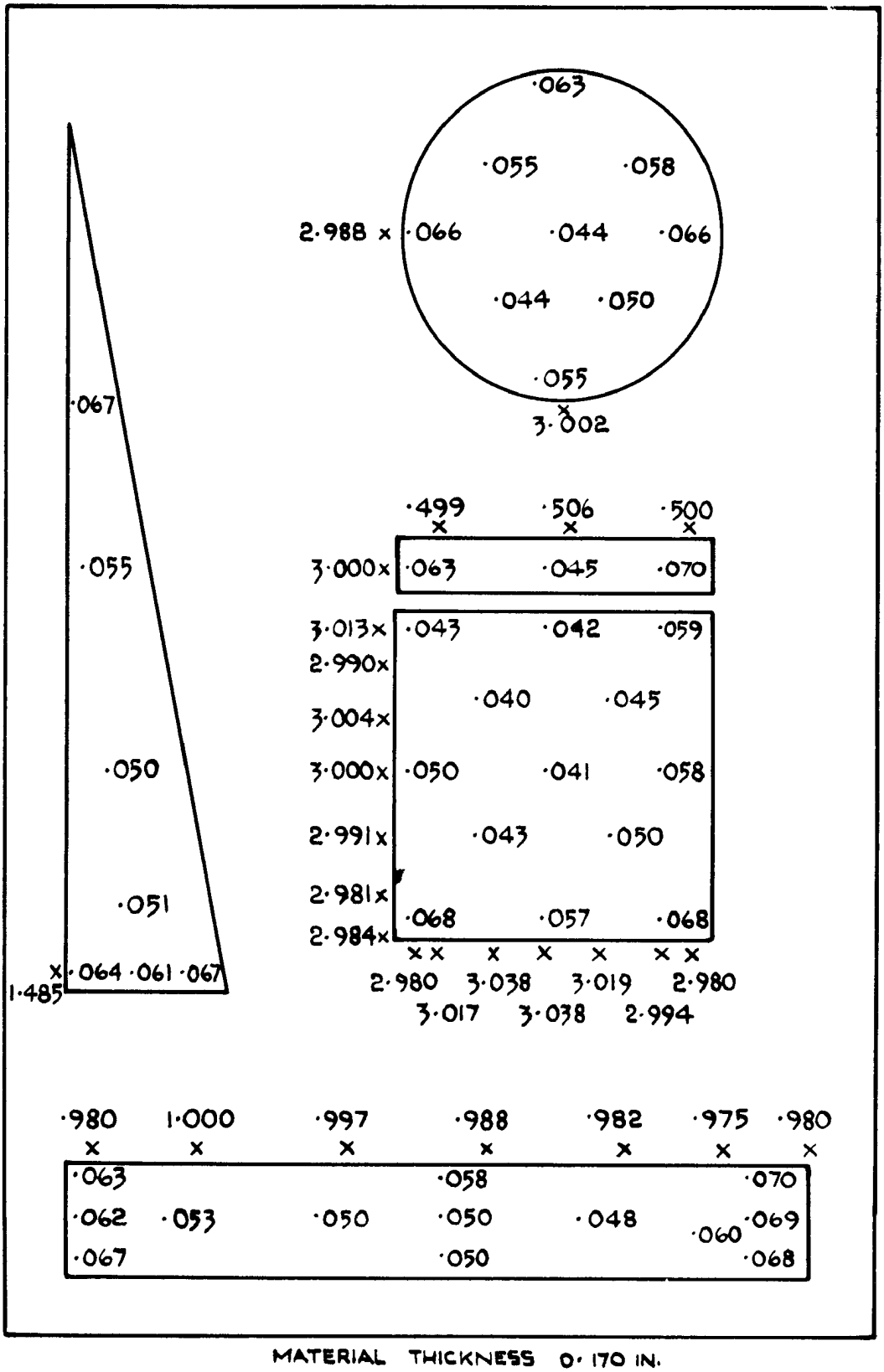


MATERIAL THICKNESS 0·170 IN.
SPECIFIED ETCH DEPTH 0·120 IN.

FINAL THICKNESS MEASUREMENTS IN INCHES.

3% CR. STEEL PANEL No. 1. DIMENSIONS AFTER CONTOURING

FIG. 20

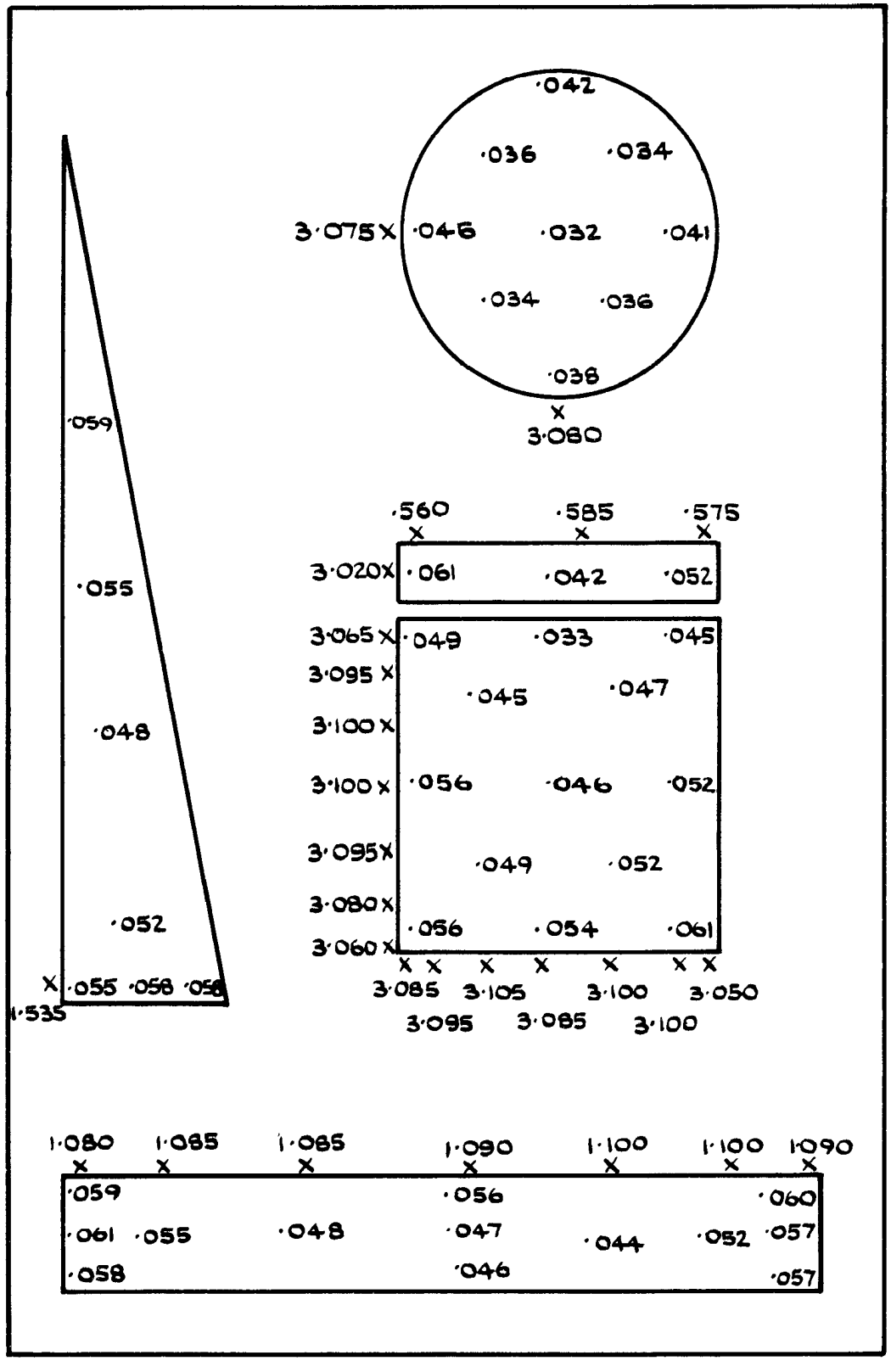


MATERIAL THICKNESS 0·170 IN.
SPECIFIED ETCH DEPTH 0·120 IN.

FINAL THICKNESS MEASUREMENTS IN INCHES.

3% C R. STEEL PANEL No. 2. DIMENSIONS AFTER CONTOURING

FIG: 21

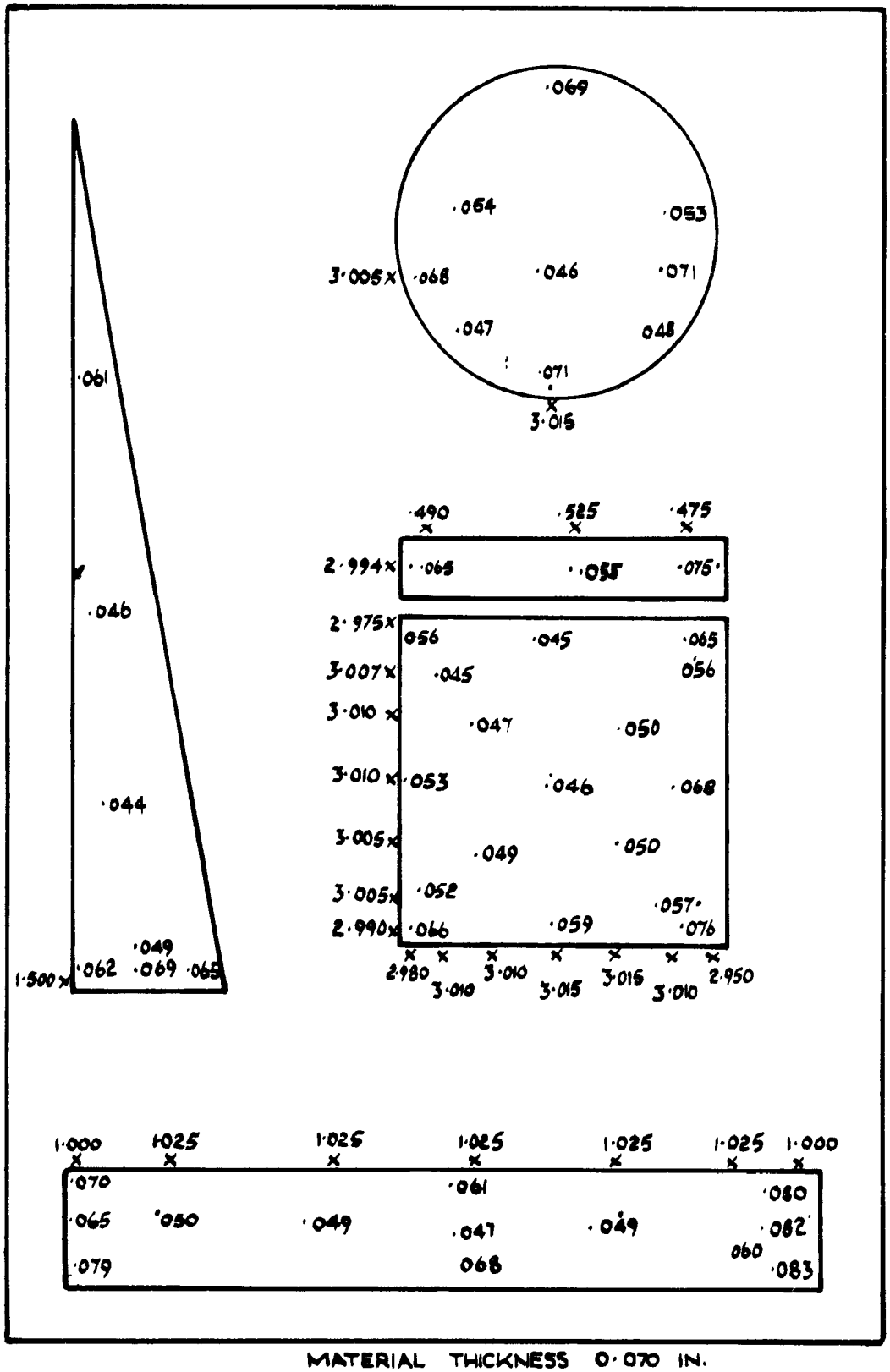


MATERIAL THICKNESS 0·170 IN.
SPECIFIED ETCH DEPTH 0·120 IN.

FINAL THICKNESS MEASUREMENTS IN INCHES

3% CR. STEEL PANEL No. 3. DIMENSIONS AFTER CONTOURING.

FIG. 22



MATERIAL THICKNESS 0·070 IN.
SPECIFIED ETCH DEPTH 0·120 IN.

FINAL THICKNESS MEASUREMENTS IN INCHES

3% CR. STEEL PANEL No. 4 DIMENSIONS AFTER CONTOURING.

FIG. 23

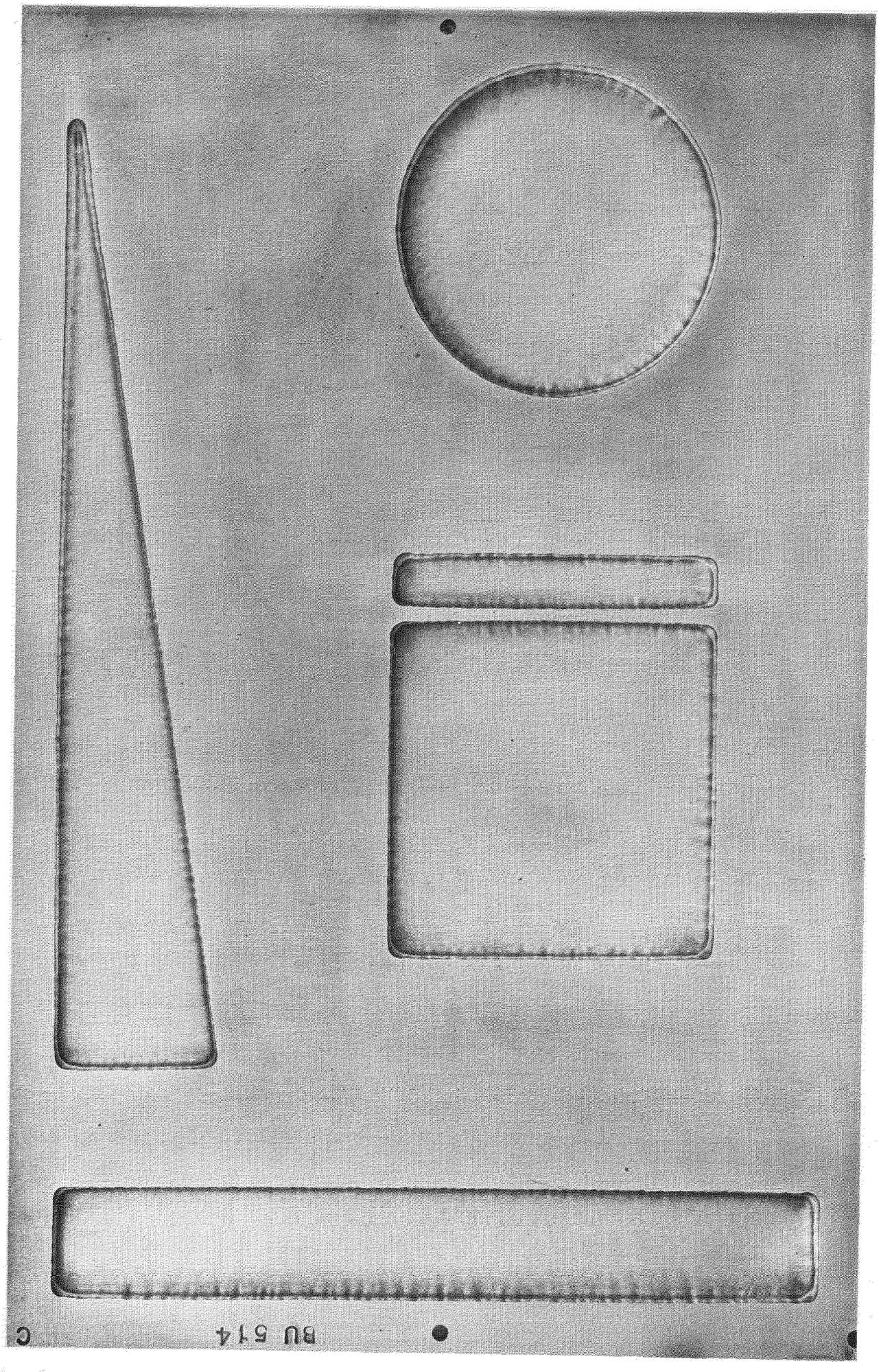
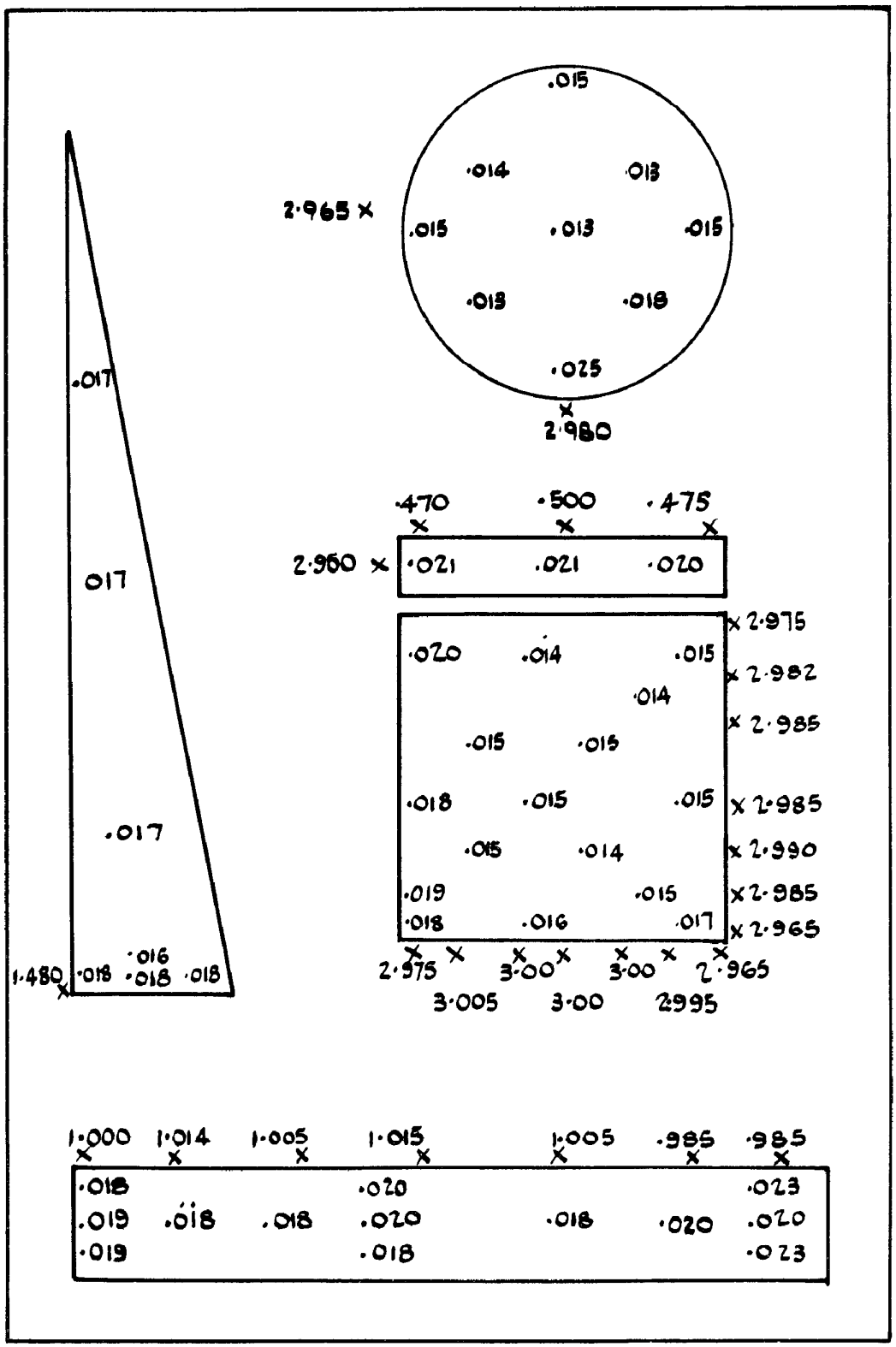


FIG. 24. R.S.140 STEEL PANEL

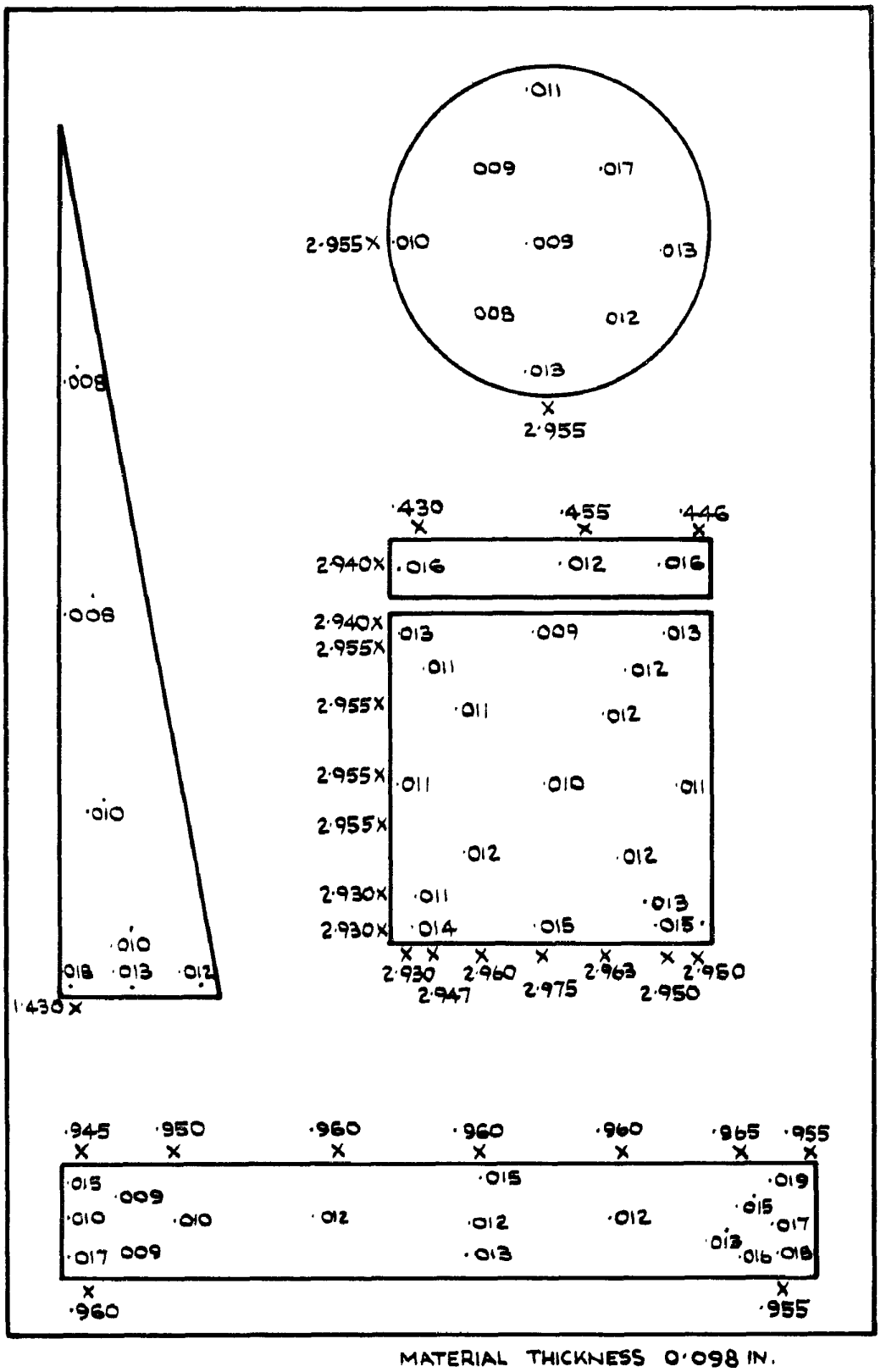


MATERIAL THICKNESS 0·098 IN.
SPECIFIED ETCH DEPTH 0·085 IN.

FINAL THICKNESS MEASUREMENTS IN INCHES.

5% C.R. STEEL PANEL No. 1. DIMENSIONS AFTER CONTOURING

FIG. 25

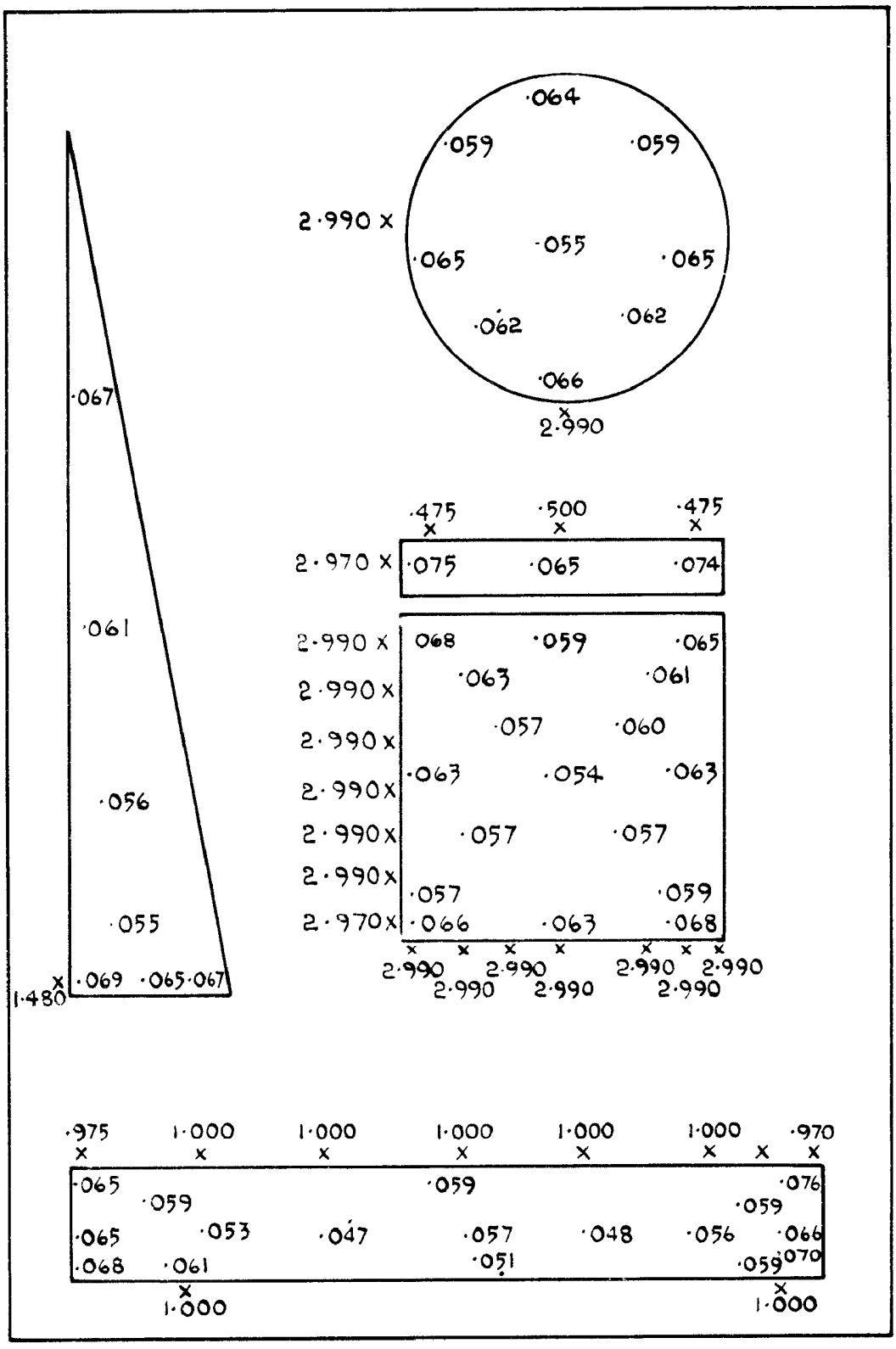


MATERIAL THICKNESS 0·098 IN.
SPECIFIED ETCH DEPTH 0·085 IN.

FINAL THICKNESS MEASUREMENTS IN INCHES.

5% CR. STEEL PANEL No. 4. DIMENSIONS AFTER CONTOURING

FIG. 26



MATERIAL THICKNESS 0·150 IN.
SPECIFIED ETCH DEPTH 0·100 IN.

FINAL THICKNESS MEASUREMENTS IN INCHES.

5% CR STEEL PANEL No. 5. DIMENSIONS AFTER CONTOURING.

FIG27

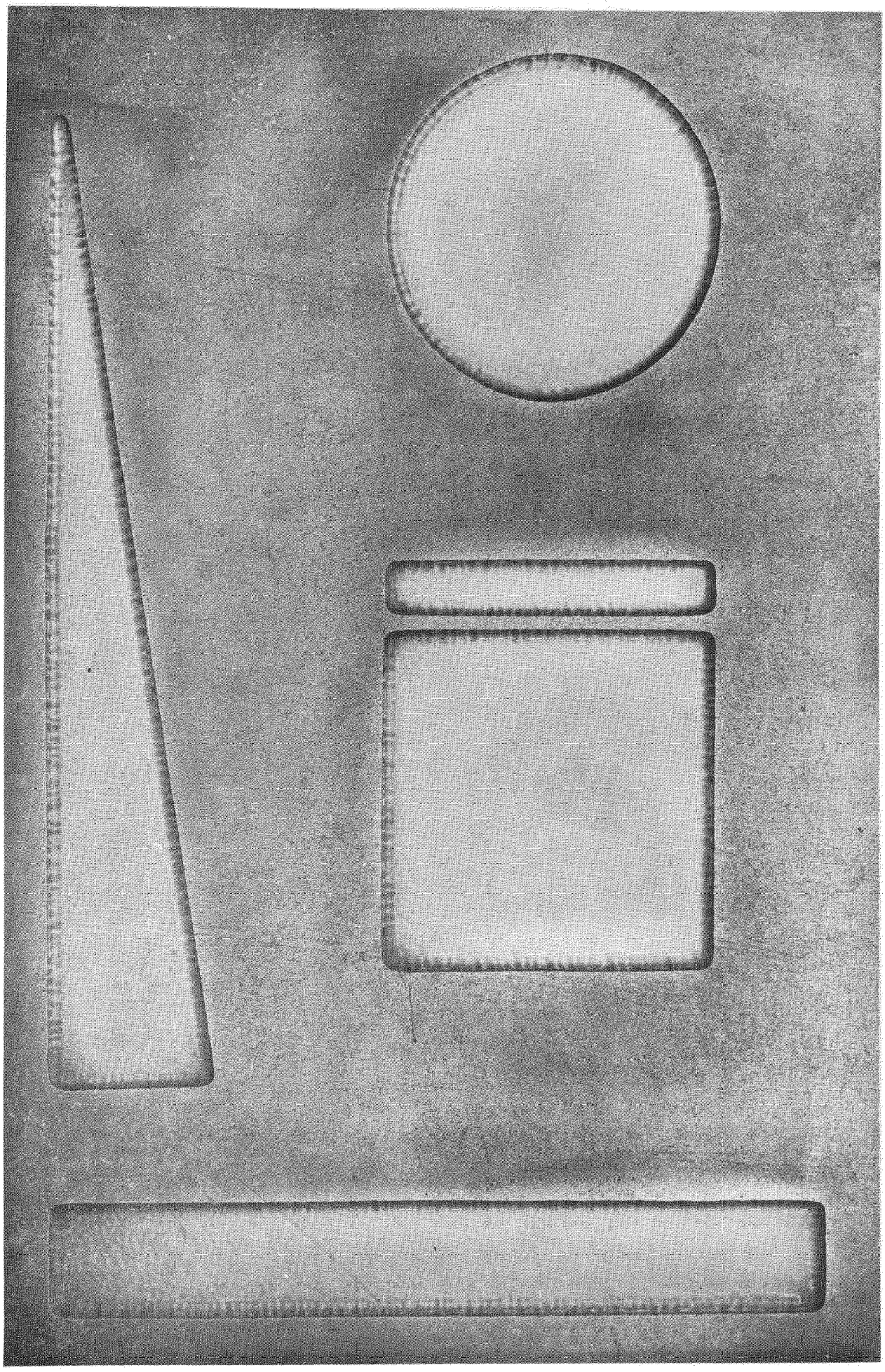


FIG. 28. H50 STEEL PANEL, No.1.

A.R.C. C.P. No.811
March, 1964.
Bristol Aerojet Ltd.

THE CHEMICAL CONTOURING OF 3% CHROMIUM - MOLYBDENUM - VANADIUM AND 5% CHROMIUM - MOLYBDENUM - VANADIUM HIGH STRENGTH STEEL SHEET

Earlier development of a chemical contouring process for 1% chromium - molybdenum steel sheet was described in S. & T. Memos 20/60 and 23/60. The present report describes the development of processes for 3% chromium-molybdenum-vanadium steel sheet and 5% chromium-molybdenum-vanadium steel sheet both heat treated to tensile strengths of not less than 105 tons/sq.in. A common etchant solution for all three steels was also developed.

contd./

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contd./

Preferred etchant compositions and proprietary masking materials are given. Etching must be carried out within fairly closely defined limits of temperature.

Specimen panels of both steels have been prepared. The surface finish was good and edge definition acceptable. All panels, however, showed considerable variation in etch depth, the depth being greater at the centres of etched pockets than near the edges.

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